QUALITY ASSURANCE PROJECT PLAN FOR THE REMEDIAL INVESTIGATION AT MIDWEST METALLICS L.P. SUMMIT, ILLINOIS U.S. EPA ID NUMBER ILD 054 348 974 REVISION 1

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Prepared by: W. Z. Baumgartner & Associates, Inc.
Prepared for: Midwest Metallics L.P.

Terence J. Coogan, Esq. - Project Coordinator	Date
J. Claiborne Thornton, III, P.E. - Consultant Project Manager	Date
Michael E. Tant, P.E. - Consultant QA Manager	Date
Theodore J. Duello, Ph.D. - Laboratory QA Manager	Date
John Gaitskill - U.S. EPA RCRA Project Manager/ Permit Writer	Date
Sherry Estes - U.S. EPA RCRA Enforcement Permitting QA Coordinator	Date

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TABLE OF CONTENTS

Page

TITLE AND SIGNATURE PAGE

TABLE OF CONTENTS

1.0 Project Description 1-1

1.1 Introduction 1-1
1.1.1 Overall Project Objectives and Decision Statements 1-1
1.1.2 Project Status/Phase 1-2
1.1.3 QAPP Preparation Guidelines . . . 1-3
1.2 Site/Facility Description 1-4
1.2.1 Location 1-4
1.2.2 Facility/Site Size and Borders . . . 1-4
1.2.3 Natural & Manmade Features . . . 1-4
1.2.4 Topography 1-4
1.2.5 Local Geology and Hydrogeology . . . 1-4
1.2.6 Surrounding Land Use 1-6
1.2.7 Ecological Communities and Habitats . . 1-6
1.3 Site/Facility History 1-6
1.3.1 General History 1-7
1.3.2 Past Data Collection Activities . . . 1-7
1.3.3 Current Status 1-7
1.4 Project Objectives and Intended Data Usages . . 1-7
1.4.1 Project Target Parameters 1-9
1.4.2 Field Parameters 1-10
1.4.3 Laboratory Parameters 1-10
1.5 Sampling Locations 1-10
1.5.1 Rationale for Selected Sampling Locations . . 1-10
1.6 Project Schedule 1-10
1.6.1 Anticipated Date of Project Mobilization . . 1-10
1.6.2 Task Bar Chart and Associated Time Frames . 1-11

2.0 Project Organization And Responsibility 2-1

2.1 Project Organization Chart 2-1

2.2 Management Responsibilities 2-1
2.3 Quality Assurance Responsibilities 2-3
2.4 Laboratory Responsibilities 2-4
2.5 Field Responsibilities 2-5
2.6 Special Training Requirements and Certifications . . 2-7
2.6.1 Training 2-7
2.6.2 Certification 2-7

3.0 Quality Assurance Objectives 3-1

3.1 Precision 3-1
3.1.1 Definition 3-1
3.1.2 Field Precision Objectives 3-1
3.1.3 Laboratory Precision Objectives . . . 3-1
3.2 Accuracy 3-2
3.2.1 Definition 3-2
3.2.2 Field Accuracy Objectives 3-2
3.2.3 Laboratory Accuracy Objectives . . . 3-2
3.3 Completeness 3-3
3.3.1 Definition 3-3
3.3.2 Field Completeness Objectives . . . 3-3
3.3.3 Laboratory Completeness Objectives . . 3-3
3.4 Representativeness 3-4
3.4.1 Definition 3-4
3.4.2 Measures to Ensure Representativeness of Field Data 3-4
3.4.3 Measures to Ensure Representativeness of Laboratory Data 3-4
3.5 Decision Rules 3-4
3.5.1 Definition 3-4
3.5.2 Decision Rule Objectives 3-5
3.6 Comparability 3-5
3.6.1 Definition 3-5
3.6.2 Measures to Ensure Comparability of Field Data . 3-5
3.6.3 Measures to Ensure Comparability of Laboratory Data 3-5
3.7 Level of Quality Control Effort 3-5

4.0 Sampling Procedures 4-1

5.0 Custody Procedures 5-1

5.1 Field Custody Procedures 5-1
5.2 Laboratory Custody Procedures 5-4
5.3 Final Evidence Files 5-5

6.0 Calibration Procedures and Frequency 6-1

6.1 Field Instrument Calibration 6-1
6.2 Laboratory Instrument Calibration 6-2

7.0 Analytical Procedures 7-1

7.1 Field Analytical Procedures 7-1
7.2 Laboratory Analytical Procedures 7-1
7.2.1 List of Project Target Compounds and Laboratory Detection Limits . . . 7-1
7.2.2 List of Associated Quality Control Samples . 7-1

8.0 Internal Quality Control Checks 8-1

8.1 Field Quality Control Checks 8-1
8.2 Laboratory Quality Control Checks 8-1

9.0 Data Reduction, Validation and Reporting 9-1

9.1 Data Reduction 9-1
9.1.1 Field Data Reduction Procedures . . . 9-1
9.1.2 Laboratory Data Reduction Procedures . . 9-1
9.2 Data Validation 9-1
9.2.1 Procedures Used to Validate Field Data . . 9-1
9.2.2 Procedures Used to Validate Laboratory Data . 9-2
9.3 Data Reporting 9-2
9.3.1 Field Data Reporting 9-2
9.3.2 Laboratory Data Reporting 9-2
9.4 Data Acquisition Requirements and Data Quality Management 9-4

10.0 Performance and System Audits and Frequency . . . 10-1
10.1 Field Performance and System Audits . . . 10-1
10.1.1 Internal Field Audits 10-1
10.1.1.1 Internal Field Audit Responsibilities 10-1
10.1.1.2 Internal Field Audit Frequency . 10-1

WZB

10.1.1.3 Internal Field Audit Procedures . 10-1
10.1.2 External Field Audits 10-2
10.1.2.1 External Field Audit Responsibilities 10-2
10.1.2.2 External Field Audit Frequency . 10-2
10.1.2.3 External Field Audit Process . 10-2
10.2 Laboratory Performance and System Audits . . 10-3
10.2.1 Internal Laboratory Audits 10-3
10.2.1.1 Internal Laboratory Audit Responsibilities 10-3
10.2.1.2 Internal Laboratory Audit Frequency 10-3
10.2.1.3 Internal Laboratory Audit Procedures 10-3
10.2.2 External Laboratory Audits 10-3
10.2.2.1 External Laboratory Audit Responsibilities 10-3
10.2.2.2 External Laboratory Audit Frequency 10-3
10.2.2.3 Overview of the External Laboratory Audit Process . . . 10-4

11.0 Preventative Maintenance 11-1

11.1 Field Instrument Preventative Maintenance . . . 11-1
11.2 Laboratory Instrument Preventative Maintenance . . 11-1
11.3 Inspection/Acceptance Requirements for Supplies and Consumables 11-2
11.3.1 Reagent Requirements 11-2
11.3.2 Reagent Storage 11-3
11.3.3 Glassware 11-4

12.0 Specific Routine Procedures Used to Evaluate Data Precision, Accuracy and Completeness 12-1

12.1 Accuracy Assessment 12-2
12.2 Precision Assessment 12-3
12.3 Completeness Assessment 12-3
12.4 Assessment of Data 12-3

13.0 Corrective Action 13-1

13.1 Field Corrective Action 13-1
13.2 Laboratory Corrective Action 13-3
13.3 Corrective Action During Data Validation and Data Assessment 13-4

14.0 Quality Assurance Reports to Management 14-1

14.1 Contents of Project QA Reports 14-1
14.2 Frequency of QA Reports 14-2
14.3 Individuals Receiving/Reviewing QA Reports . . 14-2

WZB

PROJECT DESCRIPTION

1.0 Project Description

This QAPP presents the organization, objectives, planned activities and specific Quality Assurance (QA)/Quality Control (QC) procedures associated with the Remedial Investigation (RI) for the Midwest Metallics L.P. facility in Summit, Illinois. This QAPP was written to satisfy the requirements in the order from the U.S. EPA "Requiring Monitoring, Testing, Analysis And Reporting." Specific protocols for sampling, sample handling and storage, chain of custody, and laboratory and field analysis will be described. All QA/QC procedures will be structured in accordance with applicable technical standards, U. S. EPA's requirements, regulations, guidance, and technical standards. This QAPP has been prepared in accordance with the U.S. EPA Region 5 QAPP policy as presented in *U.S. EPA RCRA QAPP Instructions*, and *The Use of Field Methods to Support RFI Streamlining*, U.S. EPA, Region 5 Memorandum, dated June 20, 1997.

1.1 Introduction

This QAPP has been prepared on behalf of Midwest Metallics L.P. by W. Z. Baumgartner & Associates, Inc. A QAPP and a Health and Safety Plan have been appended to the Remedial Investigation Work Plan, dated September 1998. A Field Sampling Procedures document has also been prepared, which has been entirely incorporated into the QAPP through specific reference.

1.1.1 Overall Project Objectives and Decision Statements

One purpose of this Remedial Investigation (RI) is to gather sufficient information to quantify risk to human health (Baseline Risk) and ecological receptors (Preliminary Ecological

WZB

Risk Assessment (PERA)) in the event that environmental contamination is determined to be present. The objectives of the RI are to determine the nature and extent of contamination at and/or migrating off-site from the facility.

Overall objectives of the data collection will be as follows:

- Characterize the Auto fluff pile to determine what portions of the pile contain hazardous waste within the meaning of Section 3001 of RCRA, 42 U.S.C. §6921;

- Characterize the potential pathways of contaminant migration from the fluff pile;

- Determine the magnitude, direction and rate of migration, and horizontal and vertical extent of any contamination within and beyond the facility boundary;

- Identify actual or potential receptors of contamination;

- Identify actual or potential receptors.

1.1.2 Project Status/Phase

During the remedial investigation, data analysis will be conducted in phases, with the results of the human health baseline risk assessment and preliminary ecological risk assessment being determining factors in decisions regarding the analysis being performed on the discrete fluff samples. The phase I analysis will include:

- Continuous sampling of soil from 10 borings until refusal, bedrock or goundwater is encountered. Analysis of soils from the six (6) borings located

adjacent to the pile will include: inorganics (13 metals plus cyanide, phenols, chloride, sulfide), volatiles, semi-volatile organics and pesticides/PCB/herbicides;

- Five sediment samples with continuous soil sampling to ten feet if sediment sample locations are accessible to equipment. Analysis to include:pH, organic carbon, and total metals.

- Eight wells are proposed for this site. Three of the wells (1 upgradient and 2 downgradient) will be analyzed for Appendix IX parameters. The remaining wells will be analyzed for: inorganics (13 metals plus cyanide, phenols, chloride, sulfide), volatiles, semi-volatile organics and pesticides/PCB/herbicides;

- Three composite fluff samples will be analyzed for inorganics (13 metals plus cyanide, phenols, chloride, sulfide), volatiles, semi-volatile organics and pesticides/PCB/herbicides, reactivity, corrosivity, ignitability and paint filter.

After analysis of the fluff composite samples in Phase I, Phase II will consist of the analysis of 40 discrete grid samples from the fluff pile for constituents of concern discovered in Phase I.

1.1.3 QAPP Preparation Guidelines

This QAPP has been prepared in accordance the *U.S. EPA Region 5 QAPP Instructions*.

1.2 Site/Facility Description

1.2.1 Location

The subject site is located at 7955 West 59th Street in the Village of Summit, Illinois.

1.2.2 Facility/Site Size and Borders

The subject site consists of approximately 35 acres and is bounded by industrial and commercial operations on all sides. The Indiana Harbor Belt Railroad right-of-way forms the south and east boundaries of the site.

1.2.3 Natural & Manmade Features

Several manmade structures are located on the subject site. An office/maintenance shop and truck scales are located near the entrance, while a scrap metal shredder and eddy current building are located along the south and east margins of the site, respectively. A storm water basin, which once served as a recirculation basin for the shredding process is located along the north property line.

1.2.4 Topography

The subject site is flat to gently sloping toward drainage ditches and/or railroad tracks which direct storm water to the north and west.

1.2.5 Local Geology and Hydrogeology

The surficial geology of the greater western Chicago area is comprised of glacial landforms, glaciolacustrine and glaciofluvial deposits, till, meltwater deposits, and recent alluvium. Till and/or alluvium is from five to two hundred feet thick and is underlain by shale, limestone, and dolomite bedrock.

Along the Des Plaines River, west of the Midwest Metallics, L.P. site, there is a major unconformity. Glacial sluiceways have cut through much of the till and Pennsylvanian rocks exposing Silurian formations of dolomite. This is a prime area for rock quarrying. The soil is thin near the river and is comprised of some alluvium overlying either till or bedrock.

The exposed or subsurface Silurian formation in the vicinity of the site is called the Racine Formation and is characterized by the Illinois Geological Survey as largely dolomite, slightly to moderately argillaceous with scattered chert nodules. Some areas contain massive to well-bedded pure dolomite. Minor beds of shale may exist at depth within this formation.

The oldest surficial deposits near the site are localized bars of sand and gravel from the Henry Formation that have in-filled glacial sluiceways. The Henry formation is largely glacial outwash containing sand and gravel with minor and local beds of silt. The outwash deposits have a thin cover of silt. Moving eastward from the Des Plaines River, the soil is derived from floors of glacial lakes flattened by wave erosion overlying glacial till.

Holocene stage alluvium is called the Cahokia Alluvium and is characterized as the deposits in floodplains and channels of modern rivers and streams containing mostly poorly sorted silt and sand with local deposits of sandy gravel.

Regional groundwater moves toward Lake Michigan. However, in areas of heavy pumping, the reverse may be true. The gradient is very small and historically, several deep rock aquifers have been used. Locally, in the Village of Summit, shallow groundwater appears to move towards the Des Plaines River and shipping canal. From the Midwest Metallics, L.P. site, this direction is to the west and northwest. During

the rainy season(s), surface water ponds mostly at the northwestern boundary of the site.

Groundwater flow direction is probably due to increasing permeability of sediments close to the river and shipping canal. Glacial lake deposits overlying till have less permeability and greater depth to bedrock. This would prohibit groundwater from moving eastward from the site.

1.2.6 Surrounding Land Use

Surrounding land use includes commercial and industrial tracts. Residential properties are found east of Archer Avenue, approximately 2,000 feet from the subject site.

1.2.7 Ecological Communities and Habitats

No critical ecological communities or habitats are known to exist on or near the subject site.

1.3 Site/Facility History

The site has been used for recycling metallic rich materials. These materials have been taken out of service for any of a variety of reasons and were sold to the Company as a commodity requiring processing. The received materials may have had liquids of different types or undesirable items removed as the initial step of processing. They were then sheared, fragmentized, torch cut, sorted mechanically or by hand, accumulated and then sold to a variety of companies for the regeneration of metals for use in industry. Those metals include rolled or cast iron, stainless steel, cast or extruded aluminum, copper, yellow and red brass, magnesium, lead, and several forms of zinc.

1.3.1 General History

Suppliers of material for the site normally arrived in trucks of varying sizes loaded with out of service, metallic rich, infeed material. After processing, the metallic portion was shipped by truck or rail to steel mills, foundries, furnaces, or facilities with advanced sortation capabilities.

1.3.2 Past Data Collection Activities

In 1988 in preparation for the acquisition of a portion of the assets of the Pielet Brothers Trading Company, the prospective purchaser installed ground water monitoring wells at several locations across the site. Other samples may have been taken.

The State of Illinois participated in the collection of samples from the storm water / recirculation pond on the properties northern border. Water and sediment samples were collected for analysis.

During a multi-modal inspection of the site by the EPA, State and City the EPA determined to collect a limited number of samples from the pile which is currently in question.

1.3.3 Current Status

At present the entire site is inactive as a metal recycling facility. Apparently there is a new owner who has determined to close the facility.

1.4 Project Objectives and Intended Data Usages

For this project, it will be necessary to gather sufficient information to evaluate the nature and extent of releases from solid waste management units, and also to determine whether unreasonable risks to human and ecological receptors are associated with the areas.

This could include evaluation of the impact of releases on human health and ecological receptors both within and beyond the facility property boundary, if applicable.

The data collection activity will specifically address the following concerns:

(A) To evaluate the impact of soil, sediment and groundwater contamination on human health risk, assuming the land use of the potentially impacted property is industrial; and

(B) To evaluate the impact of sediment, and soil contamination on ecological receptors.

Parameters listed in Tables 1.1 through 1.4 are the proposed critical measurement parameters for this project. Other constituents will be reported as the methods required to analyze the critical parameters have additional analytical capability. Screening levels for possible other contaminates will also be acquired. Analytical methods include:

- Soil - Method 6010B, 7471A , 8081A, 8082, 8260B, 8270C, 9012A, 9030A, 9251, 9066
- Sediment - Method 6010B, 7471A, Walkly Black
- Groundwater - For 8 samples - Method 6010B, 7470A , 8081A, 8082, 8260B, 8270C, 9012A, 9030A, 9251, 9066
  For 3 samples - Method 6010B, 7470A, 8081A, 8082, 8141A, 8151A, 8260B, 8270C, 8280A, 8290, 9012A, 9030A
- Fluff - Method 6010B, 7471A , 8081A, 8082, 8260B, 8270C, 9066, 9012A, 9030A, 9095A, 9251, s8.3SW-846, 1110, 1010

The other analytes to be reported are included in Appendix D.

1.4.1 Project Target Parameters

The list of target parameters for this project are PCB's, cadmium and lead. These parameters are typically detected in shredder fluff. Soil, fluff and groundwater samples will be analyzed for volatiles and semi-volatiles for additional screening of these matrix. In addition cyanide, phenols, chloride and sulfide will be analyzed in the soil, fluff and groundwater samples for screening levels. Additional parameters of reactivity, corrosivity, flash point and paint filter test will be analyzed for the composite fluff samples. To broaden the screening level for groundwater, samples from 1 upgradient and 2 downgradient wells will analyzed for Appendix IX parameters. Except for cadmium, lead and PCB, all other parameters listed in Appendix IX are not believed present in fluff at concentrations of concern. This conclusion can be made from samples obtained by W.Z. Baumgartner and Associates, Inc. after many years of sampling.

The analysis discussed above for the fluff samples will be performed on three composite samples. Parameters of concern detected during the analysis of the composite samples may be added to the list for analysis of 40 discrete samples from the fluff pile. At this time, the discrete samples taken from the fluff pile are scheduled to be analyzed for lead, cadmium, PCB and percent dry weight.

The Human Health Target Decision Levels provided in Tables 1.1 and 1.2 are based on Region V Risk-Based Screening Levels and EPA Region VI models for similar industrial facilities with proposed future industrial utilization. Table 1.3 parameters are based upon the Toxicity Characteristic Leaching Procedure (TCLP) regulatory thresholds and the leachable PCB standard contained in 40 CFR 761. Table 1.4 parameters were taken from Region V guidance.

1.4.2 Field Parameters

Field parameters to be analyzed for groundwater samples include: temperature, pH, specific conductivity, and turbidity. These parameters will be analyzed to determine if purging of the wells has stabilized the water within the sample column.

1.4.3 Laboratory Parameters

Tables 1.1 to 1.4 present the laboratory parameters of concern. Appendix D provides a list of all parameters which will be analyzed in the laboratory.

1.5 Sampling Locations

Figure 4 in the Work Plan presents the sampling locations for soil, sediment, fluff and groundwater. It is possible, however, that depending on the nature of encountered field conditions, sampling locations may be changed. The person who shall be responsible for making such decisions will be the Site Field Manager. All wells presented in Figure 4 may not be installed due to the absence of groundwater in the bore holes.

1.5.1 Rationale for Selected Sampling Locations

Rationale for selecting the sample locations is discussed in the Work Plan.

1.6 Project Schedule

1.6.1 Anticipated Date of Project Mobilization

W. Z. Baumgartner & Associates, Inc. anticipates being on site for the first stages of sample collection within fourteen days of approval of this plan and the RI Workplan. This should allow adequate time for securing needed drilling and excavating services.

1.6.2 Task Bar Chart and Associated Time Frames

The Task Bar Chart is shown in Figure 1-1.

Table 1.1- Soil Parameters of Concern

Constituent	Human Health Target Decision Level (mg/kg)	Ecological Target Decision Level (mg/kg)	Method	PQL (mg/kg)
Cadmium	850	850	6010B	1.0
Lead	1600	1600	6010B	1.0
PCB	50	50	8082	0.067*

* Aroclor dependant

Table 1.2- Sediment Parameters of Concern

Constituent	Human Health Target Decision Level (mg/kg)	Ecological Target Decision Level (mg/kg)	Method	PQL (mg/kg)
Cadmium	850	850	6010B	1.0
Lead	1600	1600	6010B	1.0
PCB	50	50	8082	0.067*

* Aroclor dependant

Table 1.3- Fluff Parameters of Concern

Constituent	TCLP Target Decision Level (mg/l)	Ecological Target Decision Level (mg/kg)	Method	PQL (mg/kg)
Cadmium	1.0	N/A	6010B	1.0
Lead	5.0	N/A	6010B	1.0
PCB	0.010	N/A	8082	0.067*

* Aroclor dependant

Table 1.4- Groundwater Parameters of Concern

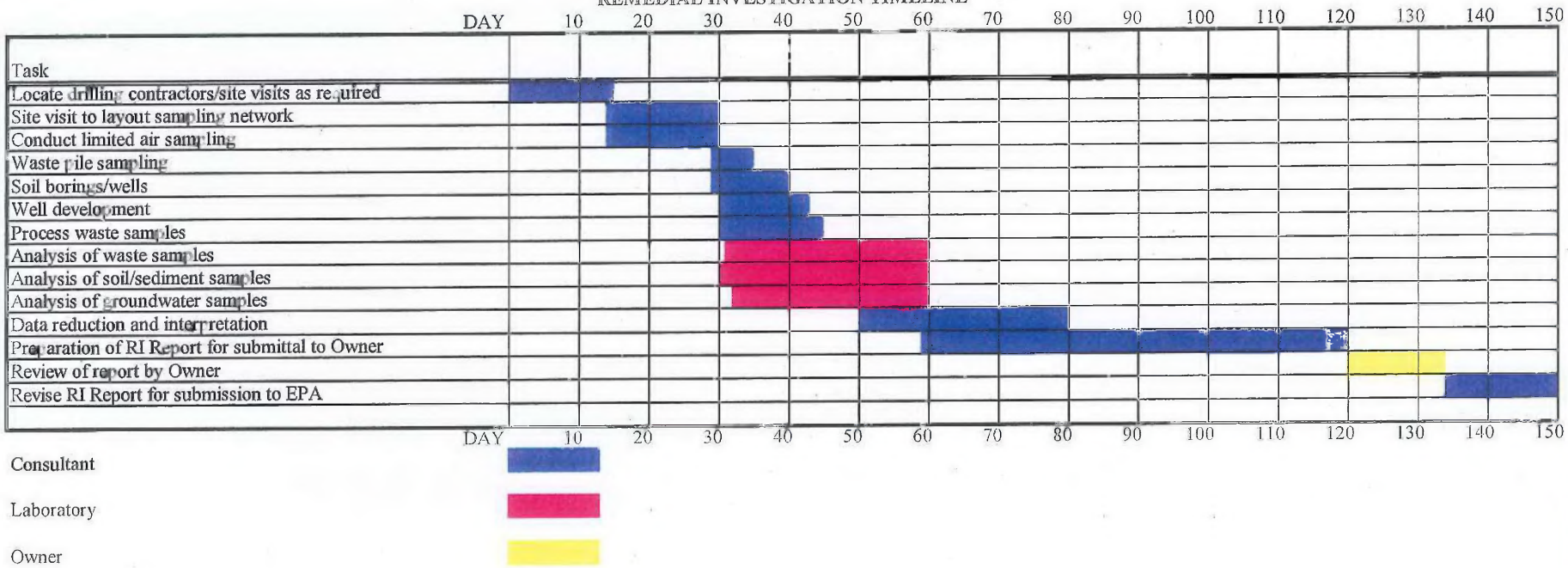
Constituent	Human Health Target Decision Level (mg/l)	Ecological Target Decision Level (mg/l)	Method	PQL (mg/l)
Cadmium	0.018	N/A	6010B	0.001
Lead	0.004	N/A	6010B	0.003
PCB	0.0087	N/A	8082	0.0005

* Aroclor dependant

The above risk-based screening levels and target decision levels will be used to determine if additional sampling analysis or remedial action is required. If soil, sediment and/or groundwater samples analyzed during this Remedial Investigation are found to contain constituents of concern at concentrations below these levels, no further assessment will be required. Similarly, should sampling and analysis of stockpiled shredder residue result in this material being designated non-hazardous and non-toxic, no further characterization will be required and Midwest Metallics L.P. and S. D. Metals, Inc. may pursue conventional management alternatives for this material.

FIGURE 1-1

MIDWEST METALLICS, L.P.
REMEDIAL INVESTIGATION TIMELINE



PROJECT ORGANIZATION AND RESPONSIBILITY

2.0 Project Organization and Responsibility

W. Z. Baumgartner & Associates, Inc. will perform the field investigation, prepare the Report and perform any subsequent studies. Project management will also be provided by W. Z. Baumgartner & Associates, Inc. The various quality assurance, field, laboratory and management responsibilities of key project personnel are defined below.

2.1 Project Organization Chart

The lines of authority specific to this investigation are presented in Figure 2-1. This chart includes all individuals discussed below.

2.2 Management Responsibilities

U.S. EPA RCRA Permit Writer/RCRA Project Manager/State Project Manager

The U.S. EPA RCRA Permit Writer (RPW)/RCRA Project Manager (RPM) has the overall responsibility for all phases of the investigation.

Owner

According to the order issued by EPA Region 5, Midwest Metallics is the "Owner" of the facility. Responsibilities include:

- Financial burden of this project along with the general partner;
- Review of findings made by the consultant;
- Responsible for implementing actions imposed on the site by the U.S. EPA upon conclusion of this investigation.

General Partner

According to the order issued by EPA Region 5, S. D. Metals, Inc. is the General

Partner in control of Midwest Metallics L.P. Responsibilities include:

- Financial burden of this project along with the owner;
- Review of findings made by the consultant;
- Responsible for implementing actions imposed on the site by the U.S. EPA upon conclusion of this investigation.

Project Coordinator

The Project Coordinator is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The Project Coordinator's primary function is to ensure that technical, financial and scheduling objectives are achieved successful. The Project Coordinator will report directly to the U.S. EPA Region 5 RPW/RPM and will provide the major point of contact and control for matters concerning the project. The Project Coordinator will:

- Acquire and apply corporate resources as needed to ensure performance within budget and schedule constraints;
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness;
- Review and analyze overall task performance with respect to planned requirements and authorizations;
- Approve all reports (deliverables) before their submission to U.S. EPA Region 5;
- Represent the project team at meetings and public hearings.

Consultant Project Manager

The Consultant Project Manager has responsibility for ensuring that the project meets U.S. EPA's objectives and quality standards. The Consultant Project Manager will provide assistance to the Project Coordinator in terms of writing and distributing the QAPP to all those parties connected with the project (including the laboratory). The Consultant Project Manager

will report directly to the Project coordinator and is responsible for technical QC and project oversight. The Consultant Project Manager will:

- Define project objectives and develop a detailed work plan schedule;
- Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task;
- Orient all field leaders and support staff concerning the project's special considerations;
- Monitor and direct the field leaders;
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product;
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness;
- Ultimately be responsible for the preparation and quality of interim and final reports.

2.3 Quality Assurance Responsibilities

The Consultant Quality Assurance (QA) Manager will remain independent of direct job involvement and day-to-day operations, and have direct access to the Consultant Project Manager as necessary, to resolve any QA dispute. He is responsible for auditing the implementation of the QA program in conformance with the demands of specific investigations, W. Z. Baumgartner and Associates, Inc. policies and U.S. EPA requirements. Specific functions and duties include:

- Performing QA audits on various phases of the field operations;
- Reviewing and approving QA plans and procedures;
- Providing QA technical assistance to project staff;
- Reporting on the adequacy, status, and effectiveness of the QA program on a

regular basis to the consultant Project Manager;

- Data validation of all samples from the analytical laboratory.

U.S. EPA RCRA Quality Assurance Coordinator (RQAC)

The U.S. EPA RQAC has the responsibility to review and approve all QAPPs. Additional U.S. EPA responsibilities for the project include:

- Conducting external Performance and System Audits of Specialized Assays Environmental, Inc.
- Evaluating results of performance evaluation sample data.
- Reviewing and evaluating analytical field and laboratory procedures.

2.4 Laboratory Responsibilities

The laboratory tasked with responsibility for analytical work is Specialized Assays Environmental, Inc., 2960 Foster Creighton Drive, Nashville, Tennessee 37204.

Laboratory Project Manager

The Laboratory Project Manager will report directly to the Consultant Project Manager and will be responsible for the following:

- Ensuring all resources of the laboratory are available on an as-required basis.
- Overseeing production and final review of analytical reports.

Laboratory Operations Manager

The Laboratory Operations Manager will report to the Laboratory Project Manager and will be responsible for:

- Coordinating laboratory analyses.
- Supervising in-house chain-of-custody.

- Scheduling sample analyses.
- Overseeing data review.
- Overseeing preparation of analytical reports.
- Approving final analytical reports prior to submission to the Consultant.

Laboratory Quality Assurance Officer

The Laboratory QA Officer has the overall responsibility for data after it leaves the laboratory. The Laboratory QA Officer will:

- Oversee laboratory QA.
- Oversee QA/QC documentation.
- Conduct detailed data review.
- Determine whether to implement laboratory corrective actions, if required.
- Define appropriate laboratory QA procedures.
- Prepare laboratory SOPs.
- Sign the title page of the QAPP.

Laboratory Sample Custodian

The Laboratory Sample Custodian will report to the Laboratory Operations Manager. Responsibilities of the Laboratory Sample Custodian will include:

- Receiving and inspecting the incoming sample containers.
- Recording the condition of the incoming sample containers.
- Signing appropriate documents.
- Verifying chain-of-custody.
- Notifying laboratory manager and laboratory supervisor of sample receipt and inspection.
- Assigning a unique identification number and customer number, and entering each into the sample receiving log.

- With the help of the laboratory manager, initiating transfer of the samples to appropriate lab sections.
- Controlling and monitoring access/storage of samples and extracts.

Laboratory Technical Staff

The laboratory technical staff will be responsible for sample analysis and identification of corrective actions. The staff will report directly to the Laboratory Operations Manager.

2.5 Field Responsibilities

Consultant Field Leader

The Consultant Project Manager will be supported by the Consultant Field Team Leader. He is responsible for leading and coordinating the day-to-day activities of the various resource specialists under his supervision. The Consultant Field Team Leader is a highly experienced environmental professional and will report directly to the Consultant Project Manager. Specific Field Team Leader responsibilities include:

- Provision of day-to-day coordination with the Consultant Project Manager on technical issues in specific areas of expertise;
- Developing and implementing of field-related work plans, assurance of schedule compliance, and adherence to management-developed study requirements;
- Coordinating and managing field staff including sampling and drilling;
- Implementing QC for technical data provided by the field staff including field measurement data;
- Adhering to work schedules provided by the Project Manager;
- Authoring, writing, and approving of text and graphics required for field team efforts;
- Coordinating and overseeing technical efforts of subcontractors assisting the

field team;

- Identifying problems at the field team level, implementing and documenting corrective action procedures, and provision of communication between team and upper management; and
- Participating in preparation of the final report.

Consultant Field Technical Staff

The technical staff will be utilized to gather and analyze data, and to prepare various task reports and support materials. All of the designated technical team members are experienced staff who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

2.6 Special Training Requirements and Certifications

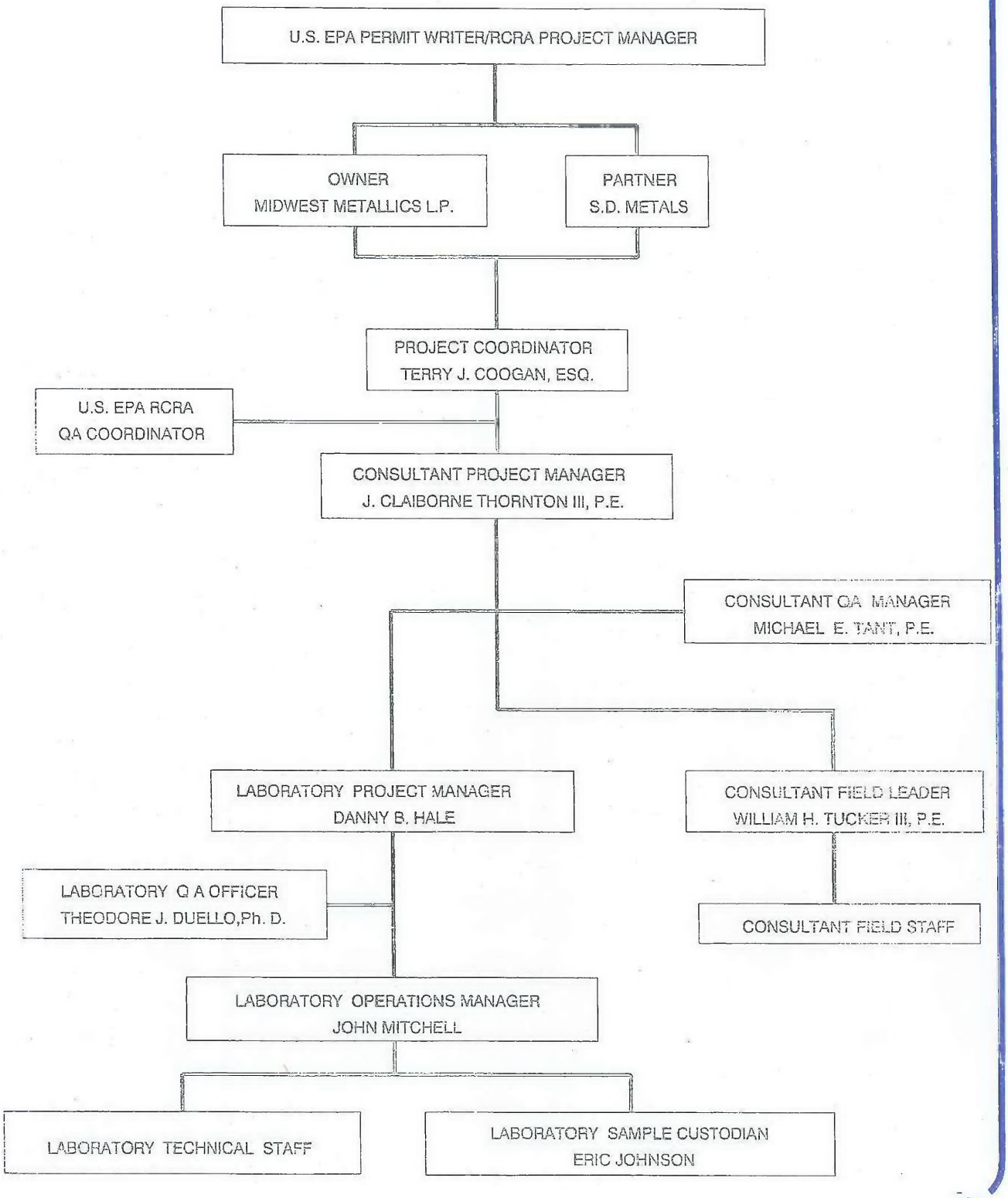
2.6.1 Training

All personnel involved with on-site operations during this investigation will have received training under the requirements of 29 CFR 1910.120, Hazardous Waste Operations and Emergency Response.

2.6.2 Certification

Certifications that each individual has received training per 29 CFR 1910.120 must be checked by the Consultant Field Leader prior to that individual entering the site.

FIGURE 2-1



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QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

3.0 Quality Assurance Objectives

The quality assurance objectives for this project is to develop and implement procedures for field sampling, laboratory analysis, chain of custody and reporting that will provide results which are legally defensible in a court of law. This section will outline the methods employed to confirm and validate the results of samples analyzed in the field and laboratory.

3.1 Precision

3.1.1 Definition

Precision is a measure of the degree to which two or more measurements are in agreement.

3.1.2 Field Precision Objectives

Obtaining duplicate samples for laboratory analysis assist in determining the precision of samples obtained in the field. Duplicates for each matrix will be collected for each 10 samples. The total number of duplicates collected for each matrix is presented in Table 3.1.

3.1.3 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of relative percent differences (RPD) and relative standard deviations (RSD) for three or more replicate samples. The equations to be used for precision in this project can be found in Section 12 of this QAPP. Precision control limits are provided in Section 5 of the laboratory's

QA Plan attached in Appendix A.

For inorganic analyses, laboratory precision shall be assessed through the analysis of a sample/sample duplicate pair and field duplicate pairs. For organic analyses, laboratory precision shall be assessed through the analysis of matrix spike/matrix spike duplicate (MS/MSD) and field duplicate samples. Note that all parameters of concern listed in Tables 1.1 through 1.4 of this QAPP are included in method spiking solutions for MS and MS/MSD analyses.

3.2 Accuracy

3.2.1 Definition

Accuracy is the degree of agreement between an observed value and an accepted reference or true value.

3.2.2 Field Accuracy Objectives

Field and trip blanks shipped to the laboratory for analysis assist in determining the accuracy of samples. Field blanks will be disguised as samples prior to being shipped to the laboratory to minimize the potential of bias in laboratory analysis. Accuracy can further be obtained through adherence to all sample handling techniques, required preservation and holding times.

3.2.3 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of MS/MSD, standard reference materials (SRM), laboratory control samples (LCS) and surrogate compounds, and the determination of percent recoveries. The equation to be used for accuracy in this project can be found in Section 12 of this QAPP. Accuracy control

limits are given in Section 5 of the laboratory's QA Plan presented in Appendix A. Note that all parameters of concern included in Tables 1.1 through 1.4 of this QAPP are included in method spiking solutions for the LCS and MS/MSD samples.

3.3 Completeness

3.3.1 Definition

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

3.3.2 Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 12 of this QAPP. The field completeness objective for this project will be greater than 90 percent.

3.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 12 of this QAPP. The laboratory completeness objective for this project, with respect to critical measurement parameters identified in Tables 1.1 through 1.4 of this QAPP, will be greater than 95 percent.

3.4 Representativeness

3.4.1 Definition

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition or an environmental condition within a defined spatial and/or temporal boundary.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness should be obtained by collecting samples at locations identified in the RI Workplan and by following collection techniques.

3.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the proper analytical procedures, appropriate methods, meeting sample holding times and analyzing and assessing field duplicate samples. The sampling network was designed to provide data representative of facility conditions. During development of this network, consideration was given to past waste disposal practices, existing analytical data, physical setting and processes, and constraints inherent to the RCRA program. The rationale of the sampling network is discussed in detail in the RI Workplan.

3.5 Decision Rules

3.5.1 Definition

A decision Rule is a statement which allows for a course of action or non-action to be taken, based on assumptions made to draw out and test its logical or empirical consequence.

3.5.2 Decision Rule Objectives

See paragraph below Table 1.4 in Section 1.0.

3.6 Comparability

3.6.1 Definition

Comparability is an expression of the confidence with which one data set can be compared to another.

3.6.2 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the field sampling procedures are followed and that proper sampling techniques are used. The RI Workplan discusses the rational behind selection of sampling location and sample type.

3.6.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented in the QAPP. Comparability is also dependent on similar QA objectives.

3.7 Level of Quality Control Effort

Field blank, trip blank, method blank, field duplicate, laboratory duplicate, laboratory control, standard reference materials (SRM) and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs.

- Field and trip blanks consisting of distilled water will be submitted to the analytical

laboratories to provide the means to assess the quality of the data resulting from the field sampling program.

- Field blank samples are analyzed to check for procedural contamination at the facility which may cause sample contamination. Field blanks will be disguised as samples to minimize the potential of bias during analysis.

- Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage.

- Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures.

- Duplicate samples are analyzed to check for sampling and analytical reproducibility.

- MS/MSDs provide information about the effect of the sample matrix on the digestion and measurement methodology. Depending on site specific circumstances, one MS/MSD should be collected for every 20 or fewer investigative samples of a given matrix. MS/MSD samples are designated/collected for organic analysis only.

One field duplicate and one field blank will be collected for every 10 or fewer samples. One trip blank consisting of distilled deionized ultra pure water will be included along with each shipment of aqueous VOC samples. The number of duplicates and field blanks cannot be determined due to the unknown depth of the borings for soil and sediment sampling.

SAMPLING PROCEDURES

4.0 Sampling Procedures

The sampling procedures to be used in this site investigation will be consistent for the objectives of this project. The Field Sampling Procedures document and the RI Workplan provide the SOP's for all sampling activities to be conducted during this investigation.

The specific field SOPs to be used are listed below.

- Groundwater Sampling Procedures
- Groundwater Monitoring Well Installation
- Well Drilling Methods
- Groundwater Sampling Equipment
- Field Analytical Procedures
- Groundwater Sampling procedures
- Groundwater Sampling Order
- Obtaining Contaminant-Free Sample Containers
- QC Sample Collection Procedures
- Sample Equipment Decontamination
- Investigation-derived Waste Management
- Discrete Soil Sampling Equipment
- Field Procedures
- Surficial Soil Sampling Procedures
- Subsoil Sampling Procedures
- Establishing a grid based sampling network

CUSTODY PROCEDURES

5.0 Custody Procedures

Custody is one of several factors which are necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including originals of all laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if:

- the item is in actual possession of a person.
- the item is in the view of the person after being in actual possession of the person.
- the item was in actual physical possession but is locked up to prevent tampering.
- the item is in a designated and identified secure area.

5.1 Field Custody Procedures

Field logbooks and field sheets will provide the means of recording data collecting activities performed during the investigation. As such, entries will be described in as much detail as possible so that persons going to the facility could reconstruct a particular situation without reliance on memory.

Field log books will be bound field survey books or notebooks. Logbooks will be assigned to field personnel. Each logbook will be identified by the project-specific number.

The title page of each logbook will contain the following:

- Person to whom the logbook is assigned.
- Logbook number.
- Project name.
- Project start date.
- End date.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection equipment being used, and the signature of the person making the entry will be entered. The names of visitors to the site, field sampling or investigation team personnel and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. All entries will be made in permanent ink, signed, and dated and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark which is initialed and dated by the sampler. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station, which includes distance measurements, shall be recorded. The number of the photographs taken of the station, if any, will also be noted. All equipment used to make measurements will be identified.

Samples will be collected following the sampling procedures documented in Section 4.0 of this QAPP. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description. The sample packaging and shipment procedures summarized below will ensure that the samples will arrive at the laboratory with the chain-of-custody intact. Examples of field custody documents and instructions for completion are presented in Appendix C of this QAPP.

- The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. Field procedures have been designed such that as few people as possible will handle the samples.

- All bottles will be identified by the use of sample tags with sample numbers, sampling locations, date/time of collection, and type of analysis.

- Sample tags will be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.

- Samples will be accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

- Samples will be properly packaged on ice at 4°C for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in and secured to the inside top of each sample box or cooler. Shipping containers will be locked and secured with strapping tape and custody seals for shipment to the laboratory. The custody seals will be attached to the front right and back left of the cooler and covered with clear plastic tape after being signed by the field team leader. The cooler will be strapped shut with strapping tape in at least two locations.

Whenever split samples are collocated with a government agency, a separate sample receipt will be prepared for those samples and marked to indicate with whom the samples are being collocated. The person relinquishing the samples to the facility or agency should request the following:

- The representatives signature acknowledging sample receipt. If the representative is unavailable or refuses to sign, this is noted in the "Received By" space.

- All shipments will be accompanied by the chain-of-custody record identifying the contents. The original record will accompany the shipment, and a copy will be retained by the sampler for returning to the sampling office.

- If the samples are sent by common carrier, a bill of lading will be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the custody form since the custody forms will be sealed inside the sample cooler and the custody seals will remain intact.

- Whenever possible, samples will be transported to the laboratory by overnight carrier.

5.2 Laboratory Custody Procedures

Laboratory custody procedures for sample receiving and log-in; sample storage and numbering; tracking during sample preparation and analysis; and storage of data are described in Section 7.0 of the laboratories Quality Assurance Plan, provided in Appendix A of this QAPP. Examples of laboratory chain-of-custody traffic reports along with instructions for

completion are included as Figure 7-1 in Appendix A of this QAPP.

5.3 Final Evidence Files

The final evidence file will be the central repository for all documents which constitute evidence relevant to sampling and analysis activities as described in this QAPP. W. Z. Baumgartner & Associates, Inc. is the custodian of the evidence file and maintains the contents of evidence files for the investigation, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews.

The final evidence file will include at a minimum:

- Field logbooks.
- Field data and data deliverables.
- Photographs.
- Drawings.
- Soil boring logs.
- Laboratory data.
- Data validation reports.
- Data assessment reports.
- Progress reports, QA reports, interim project reports, etc.
- All custody documentation (tags, forms, air bills, etc.)

CALIBRATION PROCEDURES

6.0 Calibration Procedures and Frequency

This section describes the calibration procedures and the frequency at which these procedures will be performed for both field and laboratory instruments.

6.1 Field Instrument Calibration

W. Z. Baumgartner & Associates, Inc. has the following instrumentation available for use:

- Fisherbrand Mercury Thermometers;
- Hanna Instruments Specific Conductance Meter;
- Nester Instruments pH meter;
- CP Duel pH/Conductivity Meter
- Thermo Environmental Instruments 580B Organic Vapor Analyzer
- Water Level Indicators - Slope Indicators Inc. Model #51453
- YSI Model 55 Dissolved Oxygen Meter
- Hach company Model 2100P Portable Turbidimeter

The field instruments will be calibrated as described in the field SOP's presented in the Field Sampling Procedures.

All calibration procedures performed will be documented in the field logbook and will include the date/time of calibration, name of person performing the calibration, reference standard used, temperature at which readings were taken and the readings. Multiple readings on one sample or standard, as well as readings on replicate samples, will likewise be documented.

6.2 Laboratory Instrument Calibration

Calibration procedures for a specific laboratory instrument will consist of initial calibrations (3 or 5-points), initial calibration verifications and continuing calibration verification. For a description of the calibration procedures for a specific laboratory instrument, refer to Section 9.3 of the laboratory quality assurance plan in Appendix A of this QAPP. The SOP for each analysis performed in the laboratory describes the calibration procedures, their frequency, acceptance criteria and the conditions that will require recalibration. In all cases, the initial calibration will be verified using an independently prepared calibration verification solution. The laboratory maintains a sample logbook for each instrument which will contain the following information: instrument identification, serial number, date of calibration, analyst, calibration solutions run and the samples associated with these calibrations.

ANALYTICAL PROCEDURES

7.0 Analytical Procedures

Groundwater, soil, fluff and sediment samples collected during field sampling activities for the Midwest Metallics L.P. facility investigation will be analyzed by Specialized Assays Environmental, Inc., 2960 Foster Creighton Drive, Nashville, Tennessee 37204.

7.1 Field Analytical Procedures

The procedures for field analytical determinations are provided in the SOPs presented in the Field Sampling Procedures.

7.2 Laboratory Analytical Procedures

7.2.1 List of Project Target Compounds and Laboratory Detection Limits

A complete listing of project target compounds, project quantitation limits, and current determined detection limits for each analyte group is listed in Tables 1.1 through 1.4. Method detection limits (MDLs) have been experimentally determined using procedures outlined in Appendix B of 40 CFR, part 136.

7.2.2 List of Associated Quality Control Samples

The laboratory SOP's listed in Table 7.1 include a QC section which address the minimum QC requirements for the analysis of specific analyte groups. These SOP's are located in Appendix B.

Table 7-1 SUMMARY OF ANALYTICAL PROCEDURES

ANALYTE GROUP	LAB. SOP NO.	EXTRACT. & PREP. METHOD	METHOD NUMBER
<u>Matrix: Water</u>			
Metals	44		6010B
Mercury	41		7470A
Volatiles	77	5030B	8260B
Semi-Volatiles	22 (Analysis) 24 (Prep)	3510C	8270C
Pesticides/PCB	53 (Analysis) 29 (Prep)	3510C*	8081A, 8082
Organophosphorus Pesticides	51(Analysis) 29 (Prep)	3510C	8141A
Chlorinated Herbicides	35 (Analysis) 78 (Prep)		8151A
Cyanide	14		9012A
Chloride	10,40		9251, 9056
Phenolics	55		9065
Sulfide	68		9030A
Polychlorinated Dibenzo-p-Dioxins & Polychlorinated Dibenzofurans	Contracted Out		8280A/8290

* Clean-up Methods 3620B or 3660B if necessary for PCB

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Table 7-1 (Continued) SUMMARY OF ANALYTICAL PROCEDURES

ANALYTE GROUP	LAB. SOP NO.	EXTRACT. & PREP. METHOD	METHOD NUMBER
Matrix: Soil			
Metals	44		6010B
Mercury	41		7471A
Volatiles	77	5030B	8260B
Semi-Volatiles	22 (Analysis)	3550B	8270C
	23 (Prep.)		
Pesticides/PCB	53 (Analysis)	3550B*	8081A, 8082
	28 (Prep.)		
Cyanide	14		9012A
Chloride	10,40		9251, 9056
Phenolics	55		9065
Sulfide	68		9030A

* Clean-up Methods 3620B or 3660B if necessary for PCB

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Table 7-1 (Continued) SUMMARY OF ANALYTICAL PROCEDURES

ANALYTE GROUP	LAB. SOP NO.	EXTRACT. & PREP. METHOD	METHOD NUMBER
Matrix: Fluff			
Metals (Total)	44		6010B
Metals (TCLP)	44		6010B
Metals (TCLP)	70 (Extract)		6010B
Mercury	41		7471A
Volatiles	77	5030B	8260B
Semi-Volatiles	22 (Analysis) 23 (Prep.)	3550B	8270C
Pesticides/PCB	53 (Analysis) 28 (Prep.)	3550B*	8081A, 8082
Cyanide	14		9012A
Chloride	10,40		9251, 9056
Phenolics	55		9065
Sulfide	68		9030A
Reactivity	14		s.8.3SW-846
Corrosivity	13		1110
Flash Point	30		1010
Paint Filter	52		9095A
Matrix: Sediment			
Metals	44		6010B
Mercury	41		7471A
TOC	73		Walkley Black

* Clean-up Methods 3620B or 3660B if necessary for PCB

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INTERNAL QUALITY CONTROL CHECKS

8.0 Internal Quality Control Checks

8.1 Field Quality Control Checks

QC procedures for pH, conductivity, and turbidity will include calibrations as described in Section 6.0 of the QAPP, measuring duplicate samples and checking the reproducibility of the measurements by taking multiple readings on a single sample or reference sample. Collection of the samples will be in accordance with the applicable SOPs in the Field Sampling Procedures. The number of duplicates and field blanks cannot be determined due to the unknown depth of the borings for soil and sediment sampling.

8.2 Laboratory Quality Control Checks

The laboratory identified in Section 7 of this QAPP has a QC program in place to ensure the reliability and validity of the analysis performed at the laboratory. All analytical procedures are documented in writing as SOPs and each SOP includes a QC section which addresses the minimum QC requirements for the procedure. The internal QC checks differ slightly for each individual procedure but in general the QC requirements include the following:

- Method blanks
- Reagent/preparation blanks (applicable to inorganic analysis)
- Instrument blanks
- MS/MSDs
- Surrogate spikes
- Analytical spikes (Graphite furnace)
- Laboratory duplicates
- Laboratory control standards

- Internal standard areas for GC/MS analysis
- Mass tuning for GC/MS analysis

All data obtained will be properly recorded. The data package will include a full deliverable package capable of allowing the recipient to reconstruct QC information and compare it to QC criteria. Any samples analyzed in nonconformance with the QC criteria will be reanalyzed by the laboratory, if sufficient volume is available. It is expected that sufficient volumes/weights of samples will be collected to allow for reanalysis when necessary.

DATA REDUCTION, VALIDATION AND REPORTING

9.0 Data Reduction, Validation and Reporting

9.1 Data Reduction

9.1.1 Field Data Reduction Procedures

All field data will be written into the field sampling sheets or into field log books immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Later, when the results calculation forms required for this study are being filled out, the Field Manager, identified in Section 2 of this QAPP, will review the forms to determine whether any errors have been made by the field crew.

9.1.2 Laboratory Data Reduction Procedures

The data reduction procedures are located in Section 12.1 of the Quality Assurance Plan for the laboratory included in Appendix A.

9.2 Data Validation

9.2.1 Procedures Used to Validate Field Data

The procedures to evaluate field data for this investigation include checking for transcription errors and review of field log books, on the part of field crew members. This task will be the responsibility of the Field Manager, who will otherwise not participate in making any of the field measurements, or in adding notes, data or other information to the log book.

9.2.2 Procedures Used to Validate Laboratory Data

The data validation protocols are presented in Appendix A of this QAPP. Essentially, all technical holding times shall be reviewed, instrument performance check sample results shall be evaluated, results of initial and continuing calibration will be reviewed and evaluated. Also, results of all blanks, surrogate spikes, MS/MSDs, laboratory control samples, and target compound identification and quantitation will be reviewed/evaluated by the Data Validator. One hundred percent of the analytical data shall be validated. The overall completeness of the data package will also be evaluated by the Data Validator. Completeness checks will be administered on all data to determine whether deliverables specified in the QAPP are present. At a minimum, deliverables will include sample chain-of-custody forms, analytical results, QC summaries, and supporting raw data from instrument printouts. The reviewer will determine whether all required items are present and request copies of missing deliverables.

9.3 Data Reporting

Data reporting procedures shall be carried out for field and laboratory operations as indicated below.

9.3.1 Field Data Reporting

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field, and documentation of all field calibration activities.

9.3.2 Laboratory Data Reporting

The task of reporting laboratory data (to the U.S. EPA) begins after the validation activity has been concluded. The W. Z. Baumgartner & Associates, Inc. QA

Manager must perform a final review of the report summaries and case narratives to determine whether the report meets project requirements. In addition to the record of chain-of-custody, the report format shall consist of the following:

1. Case Narrative:
   - i. Date of issuance
   - ii. Laboratory analysis performed
   - iii. Any deviations from intended analytical strategy
   - iv. Laboratory batch number
   - v. Numbers of samples and respective matrices
   - vi. QC procedures utilized and also references to the acceptance criteria
   - vii. Laboratory report contents
   - viii. Project name and number
   - ix. Condition of samples "as-received"
   - x. Discussion of whether or not sample holding times were met
   - xi. Discussion of technical problems or other observations which may have created analytical difficulties
   - xii. Discussion of any laboratory QC checks which failed to meet project criteria
   - xiii. Signature of the Laboratory QA Manager
2. Chemistry Data Package
   - i. Case narrative for each analyzed batch of samples
   - ii. Summary page indicating dates of analyses for samples and laboratory QC checks
   - iii. Cross referencing of laboratory sample to project sample identification numbers
   - iv. Description of data qualifiers to be used
   - v. Sample preparation and analyses for samples

vi. Sample results

vii. Raw data for sample results and laboratory QC samples

viii. Results of (dated) initial and continuing calibration checks, and GC/MS tuning results

ix. MS/MSD recoveries, laboratory control samples, method blank results, calibration check compounds, and system performance check compound results

x. Labeled (and dated) chromatograms/spectra of sample results and laboratory QC checks

xi. Results of tentatively identified compounds

The data package submitted will be a "CLP-like" data package consisting of all the information presented in a CLP data package (but without the CLP forms).

9.4 Data Acquisition Requirements and Data Quality Management

Hard copies and electronic copies (in .TXT format) of data will be obtained from the laboratory. Data readings gathered from the field will be input into a spreadsheet upon receipt in the office. If calculations are required, the electronic files received from the laboratory will be imported into spreadsheets to minimize the potential for error by "keying in" the data. All data input into the spreadsheets will be saved to a computer hard drive and backed up on disketts.

PERFORMANCE AND SYSTEMS AUDITS AND FREQUENCY

10.0 Performance and System Audits and Frequency

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the Field Sampling Procedures and QAPP. The audits of field and laboratory activities include two independent parts: internal and external audits.

10.1 Field Performance and System Audits

10.1.1 Internal Field Audits

10.1.1.1 Internal Field Audit Responsibilities

Internal audits of field activities including sampling, and field measurements will be conducted by the W. Z. Baumgartner & Associates, Inc. QA Manager. These audits will verify that all established procedures are being followed.

10.1.1.2 Internal Field Audit Frequency

Internal field audits will be conducted at least once at the beginning of the site sample collection activities.

10.1.1.3 Internal Field Audit Procedures

The audits will include examination of field sampling records, field screening analytical results, field instrument operating records, sample collection, handling and packaging in compliance with the established procedures, maintenance of QA procedures, chain-of-custody, etc. Follow-up

audits will be conducted to correct deficiencies, and to verify that QA procedures are maintained throughout the investigation. The audits will involve review of field measurement records, instrumentation calibration records, and sample documentation.

10.1.2 External Field Audits

10.1.2.1 External Field Audit Responsibilities

External field audits may be conducted by the U.S. EPA RCRA Permit Writer/Project Manager.

10.1.2.2 External Field Audit Frequency

External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of U.S. EPA.

10.1.2.3 External Field Audit Process

External field audits will be conducted according to the field activity information presented in the QAPP. The external field audit process can include (but not be limited to): sampling equipment decontamination procedures, sample bottle preparation procedures, sampling procedures, examination of field sampling and safety plans, sample vessel cleanliness and QA procedures, procedures for verification of field duplicates, sample preservation and preparation for shipment, as well as field screening practices.

10.2 Laboratory Performance and System Audits

10.2.1 Internal Laboratory Audits

10.2.1.1 Internal Laboratory Audit Responsibilities

The internal laboratory audit will be conducted by the Specialized Assays, Inc. QA Officer.

10.2.1.2 Internal Laboratory Audit Frequency

Internal performance audits are performed semi-annually at Specialized Assays, Inc.

10.2.1.3 Internal Laboratory Audit Procedures

The internal system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc. The laboratory audit checklist is included in Table 14-1 of Appendix A.

10.2.2 External Laboratory Audits

10.2.2.1 External Laboratory Audit Responsibilities

An external audit will be conducted as required, by appropriate QA staff of the Waste, Pesticides and Toxics Division, U.S. EPA Region 5.

10.2.2.2 External Laboratory Audit Frequency

U. S. EPA Region 5 authorities may conduct at least one audit of the

laboratory. These audits may or may not be announced and are at the discretion of the U.S. EPA.

10.2.2.3 Overview of the External Laboratory Audit Process

External audits may include any or all of: review of laboratory analytical procedures, laboratory on-site visits, and/or submission of performance evaluation samples to the laboratory for analysis. Failure of any or all audit procedures chosen can lead to laboratory disqualification, and the requirement that another suitable laboratory be chosen.

An external on-site review can consist of: sample receipt procedures, custody and sample security and log in procedures, sample through put tracking procedure, review of instrument calibration records, instrument logs and statistics (number and type), review of QA procedures, log books, sample prep procedures, sample analytical SOP review, instrument (normal or extends quantitation report) reviews, personnel interviews, review of deadlines and glassware prep, and a close out to offer potential corrective action.

It is common practice when conducting an external laboratory audit to review one or more data packages from sample lots recently analyzed by the laboratory. This review will most likely include but not be limited to;

- Comparison of resulting data to the SOP or method, including coding for deviations.
- Verification of initial and continuing calibrations within control limits.
- Verification of surrogate recoveries and instrument timing results where applicable.

- Review of extended quantitation reports for comparisons of library spectra to instrument spectra, where applicable.
- Recoveries on control standard runs.
- Review of run logs with run times, ensuring proper order of runs.
- Review of spike recoveries/QC sample data.
- Review of suspected manually integrated GC data and its cause (where applicable).
- Review of GC peak resolution for isolated compounds as compared to reference spectra (where applicable).
- Assurance that samples are run within holding times.

Ideally, the data should be reviewed while on the premises, so that any data called into question can be discussed with the staff.

PREVENTATIVE MAINTENANCE

11.0 Preventative Maintenance

11.1 Field Instrument Preventative Maintenance

The following field instrumentation will be available for use:

- Fisherbrand Mercury Thermometers;
- Hanna Instruments Specific Conductance Meter;
- Nester Instruments pH meter;
- CP Duel pH/Conductivity Meter
- Thermo Environmental Instruments 580B Organic Vapor Analyzer
- Water Level Indicators - Slope Indicators Inc. Model #51453
- YSI Model 55 Dissolved Oxygen Meter
- Hach Company Model 2100P Portable Turbidimeter

Specific preventative maintenance procedures to be followed for field equipment are based on those recommended by the manufacturer. Field instruments will be checked and calibrated daily before use. Critical spare parts such as tape and batteries will be kept on-site to reduce potential downtime. Backup instruments and equipment will be available on-site or within 1-day shipment to avoid delays in the field schedule.

11.2 Laboratory Instrument Preventative Maintenance

As part of the QA Program Plan, a routine preventative maintenance program is conducted by Specialized Assays, Inc. to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees regularly perform routine scheduled maintenance and repair of [or coordinate with the vendor for the repair of] all instruments. All maintenance that is performed is documented in the laboratory's operating record. All laboratory instruments are maintained in accordance with manufacturer's

specifications.

11.3 Inspection/Acceptance Requirements for Supplies and Consumables

All chemicals used in analysis are dated upon receipt and should not be used past the expiration data if it is listed. If no expiration date is listed, reagent chemicals are considered suitable for use for one year after it is received. Labels indication the following information on receipt and testing are to be used for critical supplies and consumables.

- Unique identification number (if not clearly shown);
- Date received;
- Date opened;
- Date tested (if performed);
- Date to be retested (if applicable);
- Expiration date.

11.3.1 Reagent Requirements

There are many different grades of analytical reagents available to the analytical chemist. All methods in use in the laboratory will specify the grade reagent that must be used in the procedure or process. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure, and, therefore, any grade reagent may be used. It is the responsibility of the analyst to carefully check the procedure and associated reagents to assure their suitability.

Reagents or working standards that are prepared in-house shall be dated, initialled by the analyst preparing the reagent and entered in to the log book for tracking purposes. The tracking procedure for all standards requires that standards be given the identification A-B-C-D-E where A represents the standard log book number, B signifies that the standard is stock, working, intermediate, spiking, or surrogate. C

represents the initials of the person preparing or receiving the standard material. D represents the page number of the log book where the entry was made, and E represents the entry number. Thus a standard may be designated 1-S-MD-59-100.

Only class "A" glassware shall be used in the preparation of any standard or reagent. Any material used in the preparation of a reagent must meet or exceed the quality of the standard or reagent chemical, e.g., solvents used in the preparation of organic standards.

All standard materials must be traceable to NBS/NIST standards, and records to that effect will be maintained in the area in which the standard is to be used.

Water used in the preparation of standards or reagents must be of laboratory grade type II. Water shall be considered type II if it has been processed through charcoal to remove organics and has passed through the laboratory reverse osmosis system and has a resulting conductance of less than 2.0 umhos. Records of the conductivity of the in-house prepared water shall be maintained. The Technical Support Manager must be immediately notified if the water exceeds specified limits. It will be the responsibility of the Technical Support Manager to notify all analytical departments of an "out of specification" situation.

The laboratory may purchase reagent grade water for use in the determination of volatile organics. This water must be certified "organic free."

11.3.2 Reagent Storage

The manner in which reagents and chemicals are stored is important from both the aspect of safety and reagent integrity. Generally, the following guidelines should

be followed:

1. Light sensitive reagents should be stored in brown glass bottles.

2. Organic stock standards will be stored in a freezer.

3. Fresh solutions of working standards will be prepared from stock solutions and will be compared to the standard being replaced before placing it into service.

11.3.3 Glassware

All volumetric glassware will be "Class A." Pyrex glass should be used where possible. For safety purposes, thick wall glassware should be used where available.

SPECIFIC ROUTINE PROCEDURES USED TO EVALUATE DATA PRECISION, ACCURACY AND COMPLETENESS

12.0 Specific Routine Procedures Used to Evaluate Data Precision, Accuracy and Completeness

The purpose of this section is to indicate the methods by which it will be ensured that the data collected for this investigation falls in line with the data quality objectives (DQOs) for the site.

Factors considered in this assessment include, but are not limited to:

- The risk assessment parameters chosen based on conditions and possible receptors involved in a project (i.e. ecological data quality levels, human health data quality levels, soil screening guidance, and the like).
- The contaminants known and/or suspected to be of concern on a project, as they relate to the data quality level parameters chosen.
- The choice of analytical and sample preparation methods for contaminants of concern, whose method detection limits will meet or exceed the data quality level concentrations for those contaminants.

Once these goals and objectives are evaluated and chosen, analytical data quality will be assessed to determine if the objectives have been met. In addition, the data will be reviewed for indications of interferences to results caused by sample matrices, cross contamination during sampling, cross contamination in the laboratory, and sample preservation and storage anomalies (i.e. samples holding time or analytical instrument problems).

12.1 Accuracy Assessment

In order to assure the accuracy of the analytical procedures, an environmental sample shall be spiked with a known amount of the analytes included in Tables 1.2 through 1.4. At a minimum, one sample spike should be included in every set of 20 samples tested on each instrument, for each sample matrix to be tested (i.e., soil, sediment, groundwater and surface water). The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the percent recovery.

Accuracy is similarly assessed by determining percent recoveries for surrogate compounds added to each field and QC sample to be analyzed for PCBs. Accuracy for the metals analysis will also
be further assessed through determination of percent recoveries for laboratory control samples, (as well as MS samples).

Percent recovery for MS/MSD results is determined according to the following equation:

$$\% R = \frac{\text{(Amount in Spiked Sample - Amount in Sample)}}{\text{Known amount added}} \times 100$$

Percent recovery for LCS and surrogate compound results is determined according to the following equation:

$$\% R = \frac{\text{Experimental Concentration}}{\text{Known amount added}} \times 100$$

12.2 Precision Assessment

The relative percent difference (RPD) between the spike and matrix spike, or matrix spike and sample duplicate in the case of metals, and field duplicate pair or laboratory duplicate pair is calculated to compare to precision DQOs and plotted. The RPD is calculated according to the following formula.

$$RPD = \frac{(\text{Amount in Sample 1} - \text{Amount in Sample 2})}{0.5(\text{Amount is Sample 1} + \text{Amount in Sample 2})} \times 100$$

12.3 Completeness Assessment

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed with a specific matrix and/or analysis. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness} = \frac{(\text{number of valid measurements})}{(\text{number of measurements planned})} \times 100$$

12.4 Assessment of Data

The field and laboratory data collected during this investigation will be used to evaluate the nature and extent of contamination at the site. Only data generated in association with QC results meeting QC objectives will be considered useable for decision making purposes.

In addition, the data obtained will be both qualitatively and quantitatively assessed on a project-wide, matrix-specific, parameter-specific and unit-specific basis. This assessment will be performed by the W. Z. Baumgartner and Associates, Inc. QA Manager and the results presented and discussed in detail in the final investigation report. Factors to be considered in this assessment of field and laboratory data will include, but not necessarily be limited to, the

following.

- Were all samples obtained using the methodologies and SOPs proposed in the QAPP?
- Were all proposed analyses performed according to the SOPs provided in the QAPP?
- Were samples obtained from all proposed sampling locations and depths?
- Do any analytical results exhibit elevated detection limits due to matrix interferences or contaminants present at high concentrations?
- Were any analytes not expected to be present at the facility, or a given unit, identified as either target parameters or Tentatively Identified Compounds (TICs)?
- Were all field and laboratory data validated according to the validation protocols, including project-specific QC objectives, proposed in the QAPP?
- Which data sets were found to be unusable (qualified as "R") based on the data validation results?
- Which data sets were found to be usable for limited purposes (qualified as "J") based on the data validation results?
- What affect do qualifiers applied as a result of data validation have on the ability to implement the project decision rules?
- Has sufficient data of appropriate quality been generated to support a human health and/or ecological screening risk assessment?
- Were the human health and/or ecological screening risk assessments conducted properly?
- Can valid conclusions be drawn for all matrices at each unit and/or area under investigation?
- Were the project-specific decision rules used as proposed during the actual

investigation?

- For any cases where the proposed procedures and/or requirements have not been met, has the affect of these issues on the project objectives been evaluated?
- Have any remaining data gaps been identified and summarized in the final investigation report?
- Based on the overall findings of the investigation and this assessment, were the original project objectives appropriately defined? If not, have revised project objectives been developed?

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CORRECTIVE ACTION

13.0 Corrective Action

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out of QC performance which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation and data assessment. All corrective action proposed and implemented should be documented in the regular QA reports to management. Corrective action should only be implemented after approval by the W. Z. Baumgartner & Associates, Inc. Project Manager, or his designee. If immediate corrective action is required, approvals secured by telephone from the W. Z. Baumgartner & Associates, Inc. Project Manager should be documented in an additional memorandum.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the W. Z. Baumgartner & Associates, Inc. Project Manager, who in turn will notify the U.S. EPA RCRA Permit Writer/Project Manager. If the problem is analytical in nature, information on these problems will be promptly communicated to the U.S. EPA RCRA Permit Writer/Project Manager. Implementation of corrective action will be confirmed in writing through the same channels.

Any nonconformance with the established QC procedures in the QAPP or Field Sampling Procedures will be identified and corrected in accordance with the QAPP. The W. Z. Baumgartner & Associates, Inc. Project Manager, or his designee, will issue a nonconformance report for each nonconformance condition.

13.1 Field Corrective Action

Corrective action in the field may be needed when the sample network is changed (i.e.

more/less samples, sampling locations other than those specified in the QAPP, etc.), sampling procedures and/or field analytical procedures require modification, etc. due to unexpected conditions. In general, the field team (technician,W. Z. Baumgartner & Associates, Inc. Project Manager and the W. Z. Baumgartner & Associates, Inc. QA Manager) may identify the need for corrective action. The field staff in consultation with the field team leader will recommend a corrective action. The W. Z. Baumgartner & Associates, Inc. Project Manager will approve the corrective measure which will be implemented by the field team. It will be the responsibility of the W. Z. Baumgartner & Associates, Inc. Project Manager to ensure the corrective action has been implemented.

If the corrective action will supplement the existing sampling plan (i.e., additional soil borings) using existing and approved procedures in the QAPP, corrective action approved by the W. Z. Baumgartner & Associates, Inc. Project Manager will be documented. If corrective actions result in less samples (or analytical fractions), alternate locations, etc., which may cause project QA objectives not to be achieved, it will be necessary that all levels of project management, including the U.S. EPA RCRA Permit Writer/Project Manager, concur with the proposed action.

Corrective action resulting from internal field audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The W. Z. Baumgartner & Associates, Inc. QA Manager will identify deficiencies and recommend corrective action to the W. Z. Baumgartner & Associates, Inc. Project Manager. Implementation of corrective actions will be performed by the W. Z. Baumgartner & Associates, Inc. Field Operations Manager and field team. Corrective action will be documented in QA reports to the entire project management.

Corrective actions will be implemented and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through

the proper channels. If corrective actions are insufficient, work may be stopped by the U.S. EPA RCRA Permit Writer/Project Manager.

If at any time a corrective action issue is identified which directly impacts project DQOs, the U.S. EPA RCRA Permit Writer/Project Manager and/or the U.S. EPA RCRA Enforcement/Permitting QA Coordinator will be notified immediately.

13.2 Laboratory Corrective Action

Corrective action in the laboratory may occur prior to, during and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, potentially high concentration samples may be identified during sample log-in or just prior to analysis.

Following consultation with lab analysts and section leaders, it may be necessary for the Specialized Assays, Inc. QC Manager to approve the implementation of corrective action. Section 13.0, paragraph two in the laboratory's QA Plan included in Appendix A of this QAPP specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures. A summary of method-specific corrective actions are found in Table 13-1 of the laboratory's QA Plan in Appendix A.

The analyst will identify the need for corrective action. The Specialized Assays, Inc. manager, in consultation with the staff, will approve the required corrective action to be implemented by the laboratory staff. The Specialized Assays, Inc. QA Manager will ensure implementation and documentation of the corrective action. If the nonconformance causes project objectives not to be achieved, it will be necessary to inform all levels of project management, including the U.S. EPA RCRA Permit Writer/Project Manager, to concur with the corrective action.

These corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both the Specialized Assays, Inc.'s corrective action log (signed by analyst, section leader and QC coordinator), and the narrative data report sent from the laboratory to the W. Z. Baumgartner & Associates, Inc. data validator. If corrective action does not rectify the situation, the laboratory will contact the W. Z. Baumgartner & Associates, Inc. Project Manager.

13.3 Corrective Action During Data Validation and Data Assessment

The facility may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory.

These actions are dependent upon the ability to mobilize the field team, whether the data to be collected is necessary to meet the required QA objectives (e.g., the holding time for samples is not exceeded, etc.). If the W. Z. Baumgartner & Associates, Inc. data assessor identifies a corrective action situation, it is the W. Z. Baumgartner & Associates, Inc. Project Manager who will be responsible for approving the implementation of corrective action, including resampling, during data assessment. All corrective actions of this type will be documented by the W. Z. Baumgartner & Associates, Inc. QA Manager.

QUALITY ASSURANCE REPORTS TO MANAGEMENT

14.0 Quality Assurance Reports to Management

The deliverables associated with the tasks identified in the Work Plan will contain separate QA sections in which data quality information collected during the task is summarized. Those reports will be the responsibility of the W. Z. Baumgartner & Associates, Inc. Project Manager and will include the Specialized Assays, Inc. QA Officer report on the accuracy, precision, and completeness of the data, as well as the results of the performance and system audits, and any corrective action needed or taken during the project.

14.1 Contents of Project QA Reports

The QA reports will contain on a routine basis, all results of field and laboratory audits, all information generated during the project reflecting on the achievement of specific DQOs, and a summary of corrective action that was implemented, and its immediate results on the project. The status of the project with respect to the Project Schedule included in the QAPP will be determined. Whenever necessary, updates on training provided, changes in key personnel will be reported. Detailed references to QAPP modifications will also be highlighted. All QA reports will be prepared in written, final format by the W. Z. Baumgartner & Associates, Inc. Project Manager or his designee. To the extent possible, assessment of the project should also be performed on the basis of available QC data and overall results in relation to originally targeted objectives.

In the event of an emergency, or in case it is essential to implement corrective action immediately, QA reports can be made by telephone to the appropriate individuals, as identified in the Project Organization and Corrective Action sections of this QAPP. However, these events, and their resolution will be addressed thoroughly in the QA report.

14.2 Frequency of QA Reports

The QA Reports will be prepared at the end of the sampling and analysis activities. The frequency of any emergency reports that must be delivered verbally cannot be estimated at the present time.

14.3 Individuals Receiving/Reviewing QA Reports

All individuals identified in the Project Organization chart will receive copies of the QA report.

APPENDIX A

LABORATORY QUALITY ASSURANCE PLAN

Revision 4.0 ***Serial Number: 4175***

Contains proprietary information. Not to be disseminated outside the organization.

Theodore J. Duello, Ph.D., QA Officer

COMPREHENSIVE QUALITY ASSURANCE PLAN

Prepared by
SPECIALIZED ASSAYS, INC.
2960 Foster Creighton Drive
Nashville, Tennessee 37204

1-800-765-0980 615-726-0177 FAX 615-726-3404

TABLE OF CONTENTS

QUALITY ASSURANCE PLAN

SECTIONS

TITLE	SECTION #	PAGES	REVSION DATE
Title Page	1.0	1	10-24-97
Table of Contents	2.0	3	10-24-97
Statement of Policy	3.0	2	10-24-97
Organization and Responsibility	4.0	7	10-24-97
Quality Assurance Objectives	5.0	53	10-24-97
Sampling Procedures	6.0	4	10-24-97
Sample Custody	7.0	8	10-24-97
Analytical Procedures	8.0	6	10-24-97
Instrument Calibration	9.0	20	10-24-97
Preventive Maintenance	10.0	5	10-24-97
Assessment of Accuracy and Precision	11.0	7	10-24-97
Data Reduction, Validation, Reporting	12.0	7	10-24-97
Corrective Action	13.0	25	10-24-97
Performance Audits	14.0	6	10-24-97
Quality Assurance Reports	15.0	1	10-24-97

TABLE OF CONTENTS, cont

TABLES

DESCRIPTION	TABLE #	SECTION	PAGES	REVISION DATE
Metallic Analytes Digestion Procedures	5-1	5	2-5	10-24-97
Sample Preparation Methods	5-2	5	6	10-24-97
Sample Cleanup Methods	5-3	5	7	10-24-97
Quality Assurance Objectives	5-4 ... 5-38	5	8-49	10-24-97
Surrogate Recovery Ranges	5-39	5	50-53	10-24-97
Sample and Preservation Requirements	6-1	6	2-4	10-24-97
Storage of Reagents and Chemicals	8-1	8	6	10-24-97
Laboratory Instrumentation	9-1	9	7-10	10-24-97
Periodic Calibration	9-2	9	11	10-24-97
Operational Calibration	9-3	9	12	10-24-97
DFTPP Key Ions and Abundance Criteria	9-4	9	13	10-24-97
BFB Key Ions and Abundance Criteria	9-5	9	14	10-24-97
Standard Sources and Preparation	9-6	9	15	10-24-97
Internal Standards for Volatile Analysis	9-7	9	16	10-24-97
Internal Standards for Semivolatile Analysis	9-8	9	17	10-24-97
Calibration Check and Perfomance Check	9-9	9	18	10-24-97
Interference Check Sample Elements Conc	9-10	9	19	10-24-97
Instrument Standardization	9-11	9	20	10-24-97
Preventive Maintenance	10-1	10	3-5	10-24-97
Quality Assurance Targets	11-1	11	7	10-24-97
Calculations	12-1	12	4	10-24-97
Data Integrity	12-2	12	5	10-24-97
Data Validation	12-3	12	6	10-24-97
Quality Control Procedures	12-4	12	7	10-24-97
Quality Control Criteria and Corrective Act	13-1	13	2-10	10-24-97
BFB Key Ion and Abundance Criteria	13-2	13	11	10-24-97
DFTPP Key Ions and Abundance Criteria	13-3	13	12	10-24-97
QC Acceptance Criteria for Method 608	13-4	13	13	10-24-97
QC Acceptance Criteria Method 8081A/8082	13-5	13	14	10-24-97
QC Acceptance Criteria for Method 625	13-6	13	15-16	10-24-97
QC Acceptance Criteria for Method 8270C	13-7	13	17-18	10-24-97
QC Acceptance Criteria for Method 624	13-8	13	19	10-24-97
QC Acceptance Criteria for Method 8260B	13-9	13	20-21	10-24-97

TABLE OF CONTENTS, cont

TABLES

DESCRIPTION	TABLE #	SECTION	PAGES	REVISION DATE
QC Acceptance Criteria for Method 601/602	13-10	13	22-23	10-24-97
QC Acceptance Criteria for Method 8021B	13-11	13	24-25	10-24-97
Internal Audit Checklist	14-1	14	2-6	10-24-97

FIGURES

DESCRIPTION	FIGURE #	SECTION	PAGES	REVISION DATE
Laboratory Organization Chart	4-1	4	7	10-24-97
Chain of Custody Form	7-1	7	8	10-24-97

3.0 STATEMENT OF POLICY

3.1 PURPOSE

As an analytical laboratory, it is our goal to produce scientific data having the highest attainable level of precision and accuracy. The quality assurance plan described in this document is a component of an overall effort to assess and control the accuracy and precision of data generated, thus making it legally and scientifically defensible.

This manual will describe management procedures and controls as well as general requirements for quality control procedures. Each method contained in our laboratory standard operating procedure manual (SOP) will have the control steps described which are specific to that method. The terms "quality assurance" and "quality control" will be defined for the purpose of this manual as follows:

QUALITY ASSURANCE: Procedures, policies, objectives, and principles which, when properly intergrated into the total laboratory system, are intended to produce data of known quality.

QUALITY CONTROL: Functional, specific actions taken by the analyst to verify processes within the laboratory.

The importance of activities described in this manual are conveyed by use of the words "must", "may", and "shall". The words "must" and "shall" indicate actions which are required. The word "may" indicates an action which is not required but is recommended. Some recommended actions may actually be required by some of the individual procedures contained in the standard operating procedures manual.

3.2 RESPONSIBILITIES AND GUIDELINES FOR PERSONNEL

The Laboratory Director is responsible for the quality of data produced, the Quality Assurance Officer is responsible for the daily monitoring of the QC program. Reporting directly to the Laboratory Director, he will make recommendations for corrective actions as needed.

All personnel will strive to meet the requirements of the QA program as it relates to their specific area of the laboratory while maintaining appropriate records to confirm that all quality control procedures are being followed. All personnel will have access to this QA manual, SOP's, and other relevant documents and are encouraged to discuss the contents with the QA Officer or Laboratory Director at any time; however, no deviations from the procedures

found herein will be made without the approval of the Laboratory Director and the laboratory QA Officer.

The laboratory QA Officer will supervise the training and evaluation of all new personnel to insure that everyone performing laboratory analyses is thoroughly familiar with the program described in this manual. Documentation relating to training of individual laboratory personnel will be kept on file in the personnel office.

4.0 ORGANIZATION AND RESPONSIBLILITY

4.1 PURPOSE

In order for the QA program to function properly all members of the staff must clearly understand and meet their individual repsonsibilities as they relate to quality control. Those responsibilities are outlined in this section and are represented graphically in Figure 4-1.

4.2 ORGANIZATIONAL CHART

Figure 4-1 illustrates the chain of responsibility within the laboratory.

4.2.1 Chief Executive Officer

The Chief Executive Officer is responsible functions relating to the laboratory as a corporation. He is the primary interface with the stockholders of the company. He has the final responsibility for the corporation.

4.2.2 Chief Financial Officer

The Chief Financial Officer is responsible for day to day operation of the business office. He is primarily responsible for accounts payable and accounts receivable.

4.4.4 President/Laboratory Director

This individual is responsible for the following:

1. Providing the general direction of the laboratory as related to systems and procedures. Authority may be delegated to appropriate individuals, however, the final responsibility lies solely with the Laboratory Director.

2. Design and implementation of the QA program.

3. In association with the QA Officer, monitor and modify when necessary the components of the QA program.

4. Laboratory chain of responsibility. This should be graphically represented by an organization chart which is updated regularly.

5. Personnel selection.

6. Logistical support operations, i.e., courriers, samplers, client service personnel.

7. Laboratory Information System design and maintenance.

8. Assist the QA Officer and the Technical Director in the review of final reports.

4.2.3 Quality Assurance Officer

This individual is responsible for the following:

1. Daily monitoring of quality control procedures.

2. Maintenance of appropriate QC charts and documents.

3. Document QC errors and work with the area supervisor to insure corrective action.

4. Initiate an in-house proficiency evaluation program involving blind samples.

5. Keep the Laboratory Director and area supervisors apprised of the general state of the quality assurance program.

6. Supervise Hazardous Waste and Health and Safety issues within the laboratory.

7. Assist in the final review of data and client reports.

8. Maintain Certifications and Validations; Report all P.E. Sample Results.

4.2.4 Technical Director

This individual will be responsible for the following:

1. Maintaining an awareness of the changes in laboratory requirements, procedures, and instrumentation.

2. Writing and updating regularly the Standard Operation Procedures (SOP) of the laboratory.

3. Monitoring all technical systems to insure proper analytical performance.

4. Interfacing with clients when questions arise relating to interpretation of data.

5. Assisting in the review of data and final laboratory reports.

4.2.5 Technical Support Manager

This individual is responsible for:

1. Direct the logging of incoming samples into the laboratory management system (LIMS).

2. Scheduling and monitoring the performance of laboratory analyses with respect to sample holding times and client expectations.

3. Direct the organized storage of hard copy raw data.

4. Generating laboratory reports and other management reports necessary for the proper function of the laboratory.

5. Manage long term storage of laboratory records.

6. Manage the organic preparation laboratory.

7. Direct electronic reporting to various clients including faxing reports.

8. Direct hard copy report distribution to clients.

4.2.6 Operations Manager

This individual is responsible for the following:

1. Management of all analytical section supervisors.

2. Assist with final review of laboratory data.

3. Supervise completion of Data Packages (e.g. CLP).

4. Insure that all daily operations specified in BOTH the SOP Manual and the Quality Assurance Manual are strictly followed.

4.2.7 Technical Services Group

These individuals are responsible for assisting Clients with technical inquiries. Incoming calls are received by Client Services; technical questions are routed to the Technical Services Group and are given to one of the following in the order listed below:

Mary Louise Lynn: primary contact for client technical inquiries.
Michael Dunn : Technical Director of laboratory
John Mitchell : Operations Manager of laboratory
Theodore Duello : Quality Assurance Officer of laboratory
Danny Hale : President / Laboratory Director

An incoming call may also be specifically routed to any one of the above at the request of the client.

4.2.8 Controller

This individual is responsible for the following:

1. Operation of the Business Office

2. Management of Client Services

3. Management of Shipping, Receiving, Sample Archives.

4.2.9 Client Services Supervisor

This individual is responsible for the following:

1. Supervising client service representatives as they form the interface between the technical departments and the client.

2. Assisting the clients in procuring the proper sampling supplies.

3. Respond to client inquiries concerning sample status.

4. Assist clients with resolution of problems concerning the Chain-of-Custody.

4.2.10 Analytical Section Supervisors

These individuals are responsible for:

1. Evaluating instruments, computer software, and personnel performance in their sections.

2. Maintenance of instrumentation such that the equipment is in proper operating condition.

3. Supervision of chemists in the section as they perform analyses to insure that SOP's are followed and that any anomalies are properly noted.

4. Performance of all proficiency elvaluations as determined by the QA officer.

5. Supervision of the daily operation of the department, e.g., personnel scheduling and workload distribution.

4.2.11 Laboratory Analysts

These individuals are responsible for the following:

1. The proper performance of all analytical procedures according to approved protocol. This will include the analysis of appropriate QC material and the final reporting of the data.

2. The analyst will report any out of control situations directly to the section supervisor.

3. The analyst will familarize himself with the manufacturers recommended maintenance schedule for instrumentation in his area and will perform and document those maintenance steps.

4.3 Required Education and Training of Technical Personnel

The following are requirements for those personnel involved in the performance of laboratory analyses:

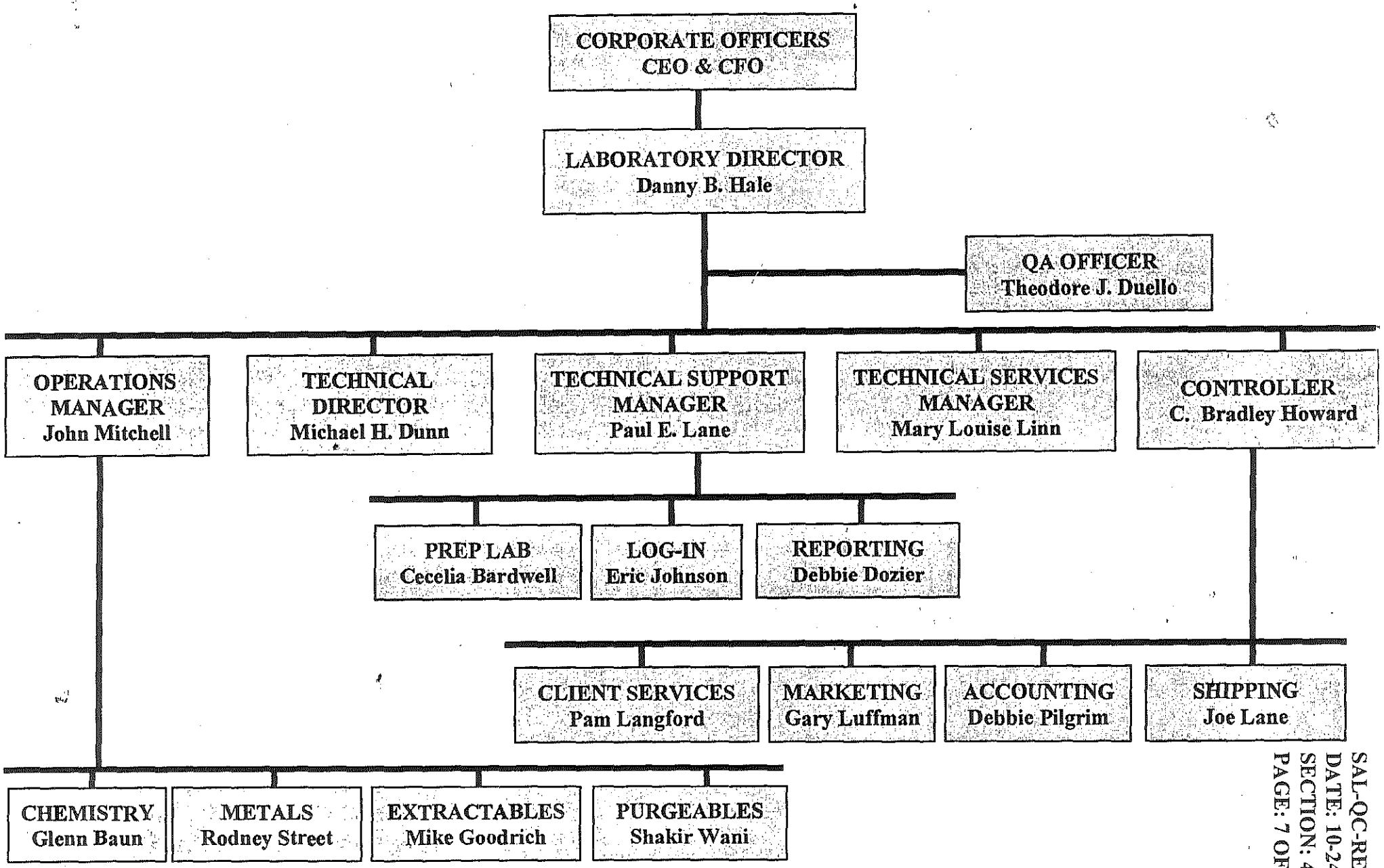
1. B.S. or B.A. degree in one of the physical sciences from an accredited college or university, plus the necessary training in our laboratory such that the QA Officer approves releasing of this employee for independent work.

2. A high school diploma, two years of college, and two years of laboratory experience in the environmental field.

3. Documentation that the analyst is capable of performing intended analyses properly to the satisfaction of the QA Officer.

ORGANIZATIONAL CHART

FIGURE 4-1



5.0 QUALITY ASSURANCE OBJECTIVES

Analytical methods used for the extraction, digestion, preparation, and analysis of organic and inorganic analytes are listed in Tables 5-1 through 5-38. Accuracy is expressed as the percent recovery within a statistically acceptable range. Precision numbers relate to the degree of correlation between duplicate pairs of samples taken through the analytical process and are expressed as a percentage. Method detection limits represent the lowest concentration of analytes that can be reported with confidence and will vary according to the matrix. The data in the Tables in this Section (Section 5) are generated from laboratory values, unless otherwise indicated. Practical Quantitation Limits or Reporting Limits are usually higher than the listed MDL's (e.g. 10 times).

TABLE 5-1

METALLIC ANALYTES DIGESTION PROCEDURES

Waters for ICP	Waters for GFAA
3005A	3020A
Aluminum	Beryllium
Antimony	Cadmium
Arsenic	Chromium
Barium	Lead
Beryllium	Thallium
Cadmium	Arsenic
Calcium	Selenium
Chromium	Antimony
Cobalt	Silver
Copper	Zinc
Iron	Copper
Lead	
Magnesium	
Manganese	
Molybdenum	
Nickel	
Potassium	
Selenium	
Silver	
Sodium	
Thallium	
Vanadium	
Zinc	

TABLE 5-1 cont

METALLIC ANALYTES DIGESTION PROCEDURES

Waters by ICP or GFAA
Microwave Digestion

3015

Aluminum
Antimony
Arsenic
Barium
Beryllium
Cadmium
Calcium
Chromium
Cobalt
Copper
Iron
Zinc
Lead
Magnesium
Manganese
Molybdenum
Nickel
Potassium
Selenium
Silver
Sodium
Thallium
Vanadium

TABLE 5-1 cont

METALLIC ANALYTES DIGESTION PROCEDURES

Soils, Sludges, Sediments
METHOD 3050 B

GFAA

Arsenic
Beryllium
Cadmium
Chromium
Lead
Selenium
Thallium
Silver
Zinc
Copper

TABLE 5-1 cont

METALLIC ANALYTES DIGESTION PROCEDURES

Microwave Digestion
Soil, Sludges, and Oils

METHOD 3051

Aluminum
Antimony
Arsenic
Boron
Barium
Beryllium
Cadmium
Calcium
Chromium
Cobalt
Copper
Iron
Lead
Magnesium
Manganese
Mercury
Molybdenum
Nickel
Potassium
Selenium
Silver
Sodium
Strontium
Thallium
Vanadium
Zinc

TABLE 5-2

SAMPLE PREPARATION METHODS

METHOD	MATRIX	FOR THESE METHODS
3510	Water	8082, 8081A, 8270C, 8310, 8100, 8151A, 8141A
3550	Soil	8082, 8081A, 8270A, 8310, 8100, 8151A, 8141A
5030	Water	8021B, 8260B, 8015B
5030	Soil	8021B, 8260B, 8015B
3540	Soil	8082, 8081A, 8270C, 8310, 8100, 8151A, 8141A
3580	Organic	8082, 8081A, 8270C, 8310, 8100, 8151A, 8141A
1311	Solid	8082, 8081A, 8270C, 8310, 8100, 8151A, 8141A, 8260B, 6010B, 7470
1312	Solid	8082, 8081A, 8270C, 8310, 8100, 8151A, 8141A, 8260B, 6010B, 7470

TABLE 5-3

SAMPLE CLEANUP METHODS

Phenols	Acids	Phthalate Esters	PAH's	Chlorinated Hydrocarbons	Base/Neutrals
3630	3650	3610	3611	3620	3650
3640		3620	3630	3640	
3650		3640	3640		

Organochlorine Pesticides	Organophosphorus Pesticides	Chlorinated Herbicides
3620	3620	8151
3640		
3660		
3665		

TABLE 5-4

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/L
200.7*	SW, WW, Eff	Aluminum	9	L	79-118	L	8.4
		Antimony	14	L	70-119	L	2.0
		Arsenic	11	L	86-121	L	3.0
		Barium	6	L	78-113	L	2.3
		Beryllium	8	L	85-122	L	0.2
		Boron	10	L	74-126	L	1.3
		Cadmium	8	L	79-119	L	0.2
		Calcium	7	L	89-110	L	1.8
		Chromium	8	L	77-119	L	1.0
		Cobalt	6	L	79-117	L	0.9
		Copper	6	L	82-115	L	0.3
		Iron	10	L	71-123	L	10.4
		Lead	11	L	78-123	L	1.4
		Magnesium	7	L	77-125	L	1.8
		Manganese	9	L	79-113	L	1.4
		Molybdenum	6	L	83-117	L	1.9
		Nickel	7	L	79-114	L	2.2
		Potassium	9	L	89-116	L	4.4
		Selenium	16	L	77-125	L	4.5
		Silver	12	L	77-129	L	0.6
		Sodium	15	L	85-115	L	28.5
		Strontium	9	L	80-109	L	0.2
		Thallium	17	L	73-119	L	2.0
		Vanadium	7	L	84-126	L	0.6
		Zinc	8	L	80-120	L	2.6

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

WW = wastewater, SW = surface water, EFF = effluents

L = Low range of the analytical curve

Method 200.7, Rev 4, May 1994 may also be used for soils; for accuracy / precision see p. 9

* Method guidelines for spike recovery: 70 - 130 %

TABLE 5-5

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL mg/kg
6010B*	SED,S HW	Aluminum	17	L	81-113	L	2.5
		Antimony	18	L	76-120	L	0.64
		Arsenic	12	L	75-118	L	1.4
		Barium	9	L	79-111	L	3.2
		Beryllium	11	L	82-115	L	0.20
		Boron	7	L	83-119	L	1.40
		Cadmium	10	L	76-118	L	0.26
		Calcium	21	L	81-114	L	2.70
		Chromium	11	L	75-120	L	1.10
		Cobalt	10	L	79-115	L	2.00
		Copper	10	L	74-122	L	0.39
		Iron	19	L	82-116	L	5.30
		Lead	10	L	75-111	L	0.35
		Magnesium	21	L	82-116	L	1.0
		Manganese	13	L	72-128	L	2.10
		Molybdenum	9	L	78-101	L	1.80
		Nickel	11	L	75-114	L	2.00
		Potassium	21	L	85-119	L	2.1
		Selenium	15	L	75-115	L	0.78
		Silver	13	L	77-122	L	0.28
		Sodium	16	L	75-122	L	3.70
		Strontium	5	L	83-118	L	0.42
		Thallium	12	L	76-116	L	1.00
		Vanadium	9	L	75-116	L	1.90
		Zinc	11	L	75-125	L	3.90

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SED = sediment, S = soil, HW = hazardous waste

L = Low range of the analytical curve

* Method guidelines for spike recovery: 75-125%

TABLE 5-6

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
6010B*	SW,GW, EFF	Aluminum	9	L	79-118	L	8.4
		Antimony	14	L	70-119	L	2.0
		Arsenic	11	L	86-121	L	3.0
		Barium	6	L	78-113	L	2.3
		Beryllium	8	L	85-122	L	0.2
		Boron	10	L	74-126	L	1.3
		Cadmium	8	L	79-119	L	0.2
		Calcium	7	L	89-110	L	1.8
		Chromium	7	L	77-119	L	1.0
		Cobalt	6	L	79-117	L	0.9
		Copper	6	L	82-115	L	0.3
		Iron	10	L	71-123	L	10.4
		Lead	11	L	78-123	L	1.4
		Lithium	8	L	77-120	L	4.0
		Magnesium	7	L	77-125	L	1.8
		Manganese	9	L	79-113	L	1.4
		Molybdenum	6	L	83-117	L	1.9
		Nickel	7	L	79-114	L	2.2
		Potassium	9	L	89-116	L	4.4
		Selenium	16	L	77-125	L	4.5
		Silver	12	L	77-129	L	0.6
		Sodium	15	L	85-115	L	28.5
		Thallium	17	L	73-119	L	2.0
		Strontium	9	L	80-109	L	0.2
		Vanadium	7	L	84-126	L	0.6
		Zinc	8	L	80-120	L	2.6

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

GW = ground water, SW = surface water, EFF = effluents

L = Low range of the analytical curve

*Method guidelines for spike recovery: 75-125%

TABLE 5-7

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL mg/l
9010	GW	Cyanide, total	12	L	70-129	L	0.002
9010		Cyanide, amenable	12	L	70-129	L	0.010
9070		Oil and Grease	17	L	83-116	L	0.420
9065		Phenolics	11	L	91-110	L	0.017
9030		Sulfide	14	L	84-104	L	0.014
9060		TOC	6	L	65-120	L	0.910
9020		TOX	10	L	78- 118	L	0.007
9056		Bromide	6	L	86-122	L	0.025
9056		Chloride	10	L	75-118	L	0.040
9056		Fluoride	5	L	83-121	L	0.030
9056		Sulfate	5	L	86-123	L	0.120
9056		Nitrate	5	L	86-113	L	0.050
9056		Nitrite	5	L	82-117	L	0.030
9040		pH	1	L	98-102	L	0.100 units

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

GW = ground water, HW = hazardous waste, SW = surface water

L = Low range of the analytical curve

TABLE 5-8

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL mg/kg
9056	SED,S,HW	Bromide	7	L	93-106	L	0.070
9056		Chloride	3	L	98-106	L	3.26
9056		Fluoride	7	L	88-107	L	0.091
9045		pH	3	L	97-104	L	0.10 Units
9056		Sulfate	6	L	93-103	L	1.41
9056		Nitrate	4	L	80-124	L	0.050
9056		Nitrite	9	L	88-119	L	0.048
9010		Cyanide	10	L	61-135	L	0.12
9010		Cyanide, ammenable	12	L	88-108	L	0.87
9071		Oil and grease	16	L	82-104	L	1.28
9065		Phenolics	11	L	71-122	L	0.19
9030		Sulfide	14	L	79-112	L	0.678
9095		Paint Filter	N/A	N/A	N/A	N/A	N/A
1010		Flash Point	N/A	N/A	N/A	N/A	N/A
1110		Corrosivity	17	L	70-130	L	1.47 mm/y
9080		Cation Exchange Cap	7	L	80-115	L	N/A
418.1 Mod		TRPH	15	L	72-127	L	1.33
9023		TOX Extracted	18	L	74-129	L	0.50
s.7.3SW-846		Reactivity	See cyanide / sulfide				
1311		TCLP	Extraction only				
1312		SPLP	Extraction only				

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SED = sediments, HW = hazardous waste, S = soil

L = Low range of the analytical curve

TABLE 5-9

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL Ug/L
7060	GW,SW	Arsenic	12	L	53-136	L	3.82
7740		Selenium	17	L	78-117	L	3.00
7761		Silver	11	L	87-124	L	0.600
7470		Mercury	10	L	78-125	L	0.072
7041		Antimony	9	L	80-98	L	5.40
7131		Cadmium	8	L	87-113	L	0.380
7191		Chromium	12	L	77-109	L	0.400
7841		Thallium	10	L	84-119	L	2.00
7421		Lead	9	L	60-136	L	1.30
7196		Chromium +6	12	L	88-129	L	3.70

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

GW = ground water, HW = hazardous waste

L = Low range of the analytical curve

TABLE 5-10

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
206.2	SW, GW,	Arsenic	12	L	83-121	L	3.82
270.2	EFF	Selenium	17	L	72-127	L	3.00
272.2		Silver	11	L	87-124	L	0.600
245.2		Mercury	10	L	73-129	L	0.072
204.2		Antimony	9	L	80-98	L	5.40
213.2		Cadmium	8	L	77-143	L	0.380
218.2		Chromium	12	L	74-123	L	0.400
279.2		Thallium	10	L	87-115	L	2.00
239.2		Lead	9	L	70-123	L	1.30
218.4		Chromium +6	12	L	58-130	L	3.70

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

GW = ground water, SW = surface water, EFF = effluent

L = Low range of the analytical curve

TABLE 5-11

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL mg/kg
7060	S,SED	Arsenic	15	L	67-118	L	0.640
7740	HW	Selenium	20	L	64-111	L	0.600
7761		Silver	11	L	78-96	L	0.120
7471		Mercury	20	L	67-138	L	0.021
7131		Cadmium	11	L	89-112	L	0.076
7191		Chromium	14	L	76-126	L	0.130
7841		Thallium	10	L	88-112	L	0.620
7421		Lead	18	L	58-134	L	0.260
7041		Antimony	17	L	65-130	L	0.592

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

S = soil, SED = sediment

L = Low range of the analytical curve

TABLE 5-12

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL mg/l
335.3	SW,EFF	Cyanide, total	12	L	70-129	L	0.004
335.1		Cyanide, ammenable	12	L	70-129	L	0.010
413.1		Oil and Grease	39	L	83-116	L	0.420
413.2		Oil and Grease	8	L	92-110	L	0.036
1664		Oil and Grease	15	L	82-119	L	1.000
360.1		Dissolved Oxygen	9	L	95-105	L	0.10
420.1		Phenolics	11	L	71-122	L	0.017
370.1		Silica	20	L	80-120	L	0.090
120.1		Conductance	4	L	95-106	L	0.06uMho/cm
376.2		Sulfide	11	L	79-117	L	0.020
377.1		Sulfite	19	L	82-116	L	2.97
425.1		Detergents (MBAS)	8	L	74-114	L	0.050
170.1		Temperature	10	L	95-105	L	N/A
140.1		Odor	N/A	L	N/A	L	N/A
350.3		Ammonia Nitrogen	8	L	78-114	L	0.091
351.4		Kjeldahl Nitrogen	10	L	55-140	L	0.028
353.3		Nitrate Nitrogen	9	L	85-115	L	0.066
353.3		Nitrate/Nitrite Nitrogen	9	L	85-115	L	0.020
354.1		Nitrite Nitrogen	9	L	87-112	L	0.006
365.2		Ortho Phosphorus	7	L	74-130	L	0.018
365.1		Phosphorus	9	L	71-127	L	0.026
405.1		BOD	20	L	70-95	L	2.56
SM-5210		BOD	20	L	70-95	L	2.20
SM-5210		CBOD	20	L	70-95	L	2.19
410.4		COD	17	L	84-123	L	1.93
415.1		TOC	6	L	65-120	L	0.91
418.1		TRPH	13	L	83-126	L	0.050
SM-9222B		Total Coliform	N/A	N/A	N/A	N/A	N/A
SM-9222D		Total Coliform	N/A	N/A	N/A	N/A	N/A
418.1 Arizona		TRPH	12	L	84-116	L	0.050

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SW = surface water, GW = ground water, EFF = effluent

L = Low range of the analytical curve

qa5tb4e.

TABLE 5-13

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL mg/l
305.1	SW,GW,EFF	Acidity	5	L	95-105	L	1.00
310.1		Alkalinity	3	L	96-105	L	1.53
300		Bromide	6	L	86-122	L	0.025
325.1		Chloride	4	L	90-110	L	0.380
300		Chloride	10	L	75-118	L	0.040
110.2		Color	8	L	N/A	L	N/A
340.2		Fluoride	9	L	86-116	L	0.021
300		Fluoride	5	L	83-121	L	0.030
130.2		Hardness	5	L	93-103	L	1.34
150.1		pH	1	L	98-102	L	0.10
160.3		Solids, total	11	L	85-115	L	8.56
160.1		Solids, filterable	11	L	85-115	L	19.1
160.2		Solids, non-filterable	11	L	85-115	L	7.8
160.5		Solids, settleable	10	L	90-110	L	0.095
160.4		Solids, volatile	14	L	80-120	L	1.0 %
375.4		Sulfate	10	L	76-133	L	4.09
300		Sulfate	5	L	86-123	L	0.120
353.3		Nitrate	7	L	85-119	L	0.007
300		Nitrate	5	L	86-113	L	0.050
300		Nitrite	5	L	82-117	L	0.030
180.1		Turbidity	13	L	84-114	L	0.088
330.5		Chlorine, residual	11	L	92-107	L	0.009

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SW = surface water, GW = ground water, EFF = effluent

L = Low range of the analytical curve

qa5tb4c.

TABLE 5-14

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
8015B	SW,GW	TRPH High	24	L	59-124	L	20.0
		TRPH Low	20	L	69-127	L	21.3
8021B Partial List	SW,GW	Benzene	17	L	84-118	L	0.17
		Toluene	17	L	80-121	L	0.24
		Ethyl benzene	17	L	80-125	L	0.15
		o-Xylene	17	L	81-127	L	0.25
		m,p-Xylene	17	L	78-132	L	0.48
		MTBE	16	L	68-124	L	0.78
		Naphthalene	27	L	72-131	L	0.94
		IPE	19	L	77-130	L	0.64
		Chlorobenzene	15	L	82-125	L	0.24
		1,2-Dichlorobenzene	18	L	76-120	L	0.38
		1,3-Dichlorobenzene	14	L	80-121	L	0.35
		1,4-Dichlorobenzene	16	L	84-115	L	0.18
8151A	SW,GW	2,4-D	14	L	38-153	L	0.60
		2,4-DB	28	L	28-159	L	0.84
		2,4,5-T	29	L	26-159	L	0.10
		2,4,5-TP	22	L	27-151	L	0.06
		Dalapon	40	L	20-107	L	2.73
		Dicamba	32	L	21-143	L	0.05
		Dichloroprop	40	L	29-142	L	0.34
		Dinoseb	28	L	31-158	L	0.05
		MCPA	41	L	33-164	L	43
		MCPP	42	L	40-150	L	44
8011/ 504.1	SW,GW	Ethylene Dibromide	25	L	74-122	L	0.015
		1,2-Dibromo-3-Chloropropane	17	L	79-131	L	0.017

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SW = surface water, GW = ground water

L = Low range of the analytical curve

TABLE 5-15

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/kg
8021B	S,SED,HW	Benzene	18	L	81-114	L	0.24
Partial		Toluene	18	L	81-116	L	0.26
List		Ethyl benzene	19	L	81-119	L	0.22
		o-Xylene	18	L	83-120	L	0.15
		m,p-Xylene	18	L	83-121	L	0.44
		MTBE	20	L	60-139	L	3.90
		IPE	19	L	76-136	L	3.80
		Naphthalene	27	L	60-138	L	0.70
8015	S,SED,HW	TRPH High	29	L	51-123	L	51.0
		TRPH Low	21	L	62-129	L	191
8151A	S,SED,HW	2,4-D	32	L	39-138	L	54
		2,4-DB	60	L	19-152	L	84
		2,4,5-T	19	L	35-133	L	8.5
		2,4,5-TP	57	L	13-143	L	5.0
		Dalapon	52	L	10-101	L	331
		Dicamba	26	L	19-130	L	12.7
		Dichloroprop	22	L	37-130	L	47.4
		MCPA	25	L	30-136	L	8050
		MCPP	23	L	38-137	L	6860
		Dinoseb	59	L	22-105	L	54

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SED = sediments, S = soil, HW = hazardous waste

L = Low range of the analytical curve

TABLE 5-16

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
615	SW,GW	2,4-D	14	L	38-153	L	0.60
		2,4-DB	28	L	28-159	L	0.84
		2,4,5-T	29	L	26-159	L	0.10
		2,4,5-TP	22	L	27-151	L	0.06
		Dalapon	40	L	20-107	L	2.73
		Dicamba	32	L	21-143	L	0.05
		Dichloroprop	40	L	29-142	L	0.34
		Dinoseb	28	L	31-158	L	0.05
		MCPA	41	L	33-164	L	43
		MCPP	42	L	40-150	L	44
8082	GW,SW	PCB 1016	15	L	78-164	L	0.0117
		PCB 1221	31	L	57-133	L	0.0394
		PCB 1232	30	L	26-135	L	0.0142
		PCB 1242	26	L	62-144	L	0.0208
		PCB 1248	16	L	56-150	L	0.0265
		PCB 1254	14	L	66-140	L	0.0212
		PCB 1260	26	L	60-141	L	0.0254

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

GW = Ground water, SW = surface water

L = Low range of the analytical curve

TABLE 5-17

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/kg
8082	S,SED,HW	PCB 1016	50	L	31-122	L	18.1
		PCB 1221	40	L	61-146	L	22.6
		PCB 1232	38	L	30-141	L	18.0
		PCB 1242	48	L	26-171	L	29.1
		PCB 1248	36	L	27-139	L	20.1
		PCB 1254	33	L	29-141	L	13.0
		PCB 1260	29	L	34-144	L	12.6

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SED = sediments, HW = hazardous waste, S = soil

L = Low range of the analytical curve

TABLE 5-18

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
602	SW,EFF	Benzene	17	L	84-118	L	0.17
		Toluene	17	L	80-121	L	0.24
		Ethyl benzene	17	L	80-125	L	0.15
		o-Xylene	17	L	81-127	L	0.25
		m,p-Xylene	17	L	78-132	L	0.48
		MTBE	16	L	68-124	L	0.78
		Naphthalene	27	L	72-131	L	0.94
		IPE	19	L	77-130	L	0.64
		Chlorobenzene	15	L	81-125	L	0.24
		1,2-Dichlorobenzene	18	L	76-120	L	0.38
		1,3-Dichlorobenzene	14	L	80-121	L	0.35
		1,4-Dichlorobenzene	16	L	84-115	L	0.18

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SW = surface water, EFF = effluents

L = Low range of the analytical curve

TABLE 5-19

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
601	SW, EFF	Dichlorodifluoromethane	13	L	66-109	L	0.47
		Chloromethane	13	L	67-116	L	0.92
		Vinyl chloride	10	L	74-120	L	0.47
		Bromomethane	11	L	72-109	L	0.81
		Chloroethane	11	L	77-114	L	0.91
		Trichlorofluoromethane	10	L	72-122	L	0.67
		1,1-Dichloroethene	11	L	81-119	L	0.59
		Methylene chloride	12	L	73-128	L	0.35
		t-1,2-Dichloroethene	13	L	83-115	L	0.35
		1,1-Dichloroethane	18	L	74-121	L	0.24
		c-1,2-Dichloroethene	10	L	82-116	L	0.25
		Bromochloromethane	10	L	73-118	L	0.24
		Chloroform	11	L	81-121	L	0.28
		2,2-Dichloropropane	19	L	87-116	L	0.44
		1,2-Dichloroethane	17	L	73-130	L	0.24
		1,1,1-Trichloroethane	16	L	79-116	L	0.34
		1,1-Dichloropropene	19	L	88-108	L	0.18
		Carbon tetrachloride	16	L	77-118	L	0.24
		Dibromomethane	10	L	77-114	L	0.22
		1,2-Dichloropropane	18	L	79-121	L	0.17
		Trichloroethene	13	L	79-123	L	0.25
		Bromodichloromethane	18	L	73-128	L	0.17
		c-1,3-Dichloropropene	12	L	74-117	L	0.22
		t-1,3-Dichloropropene	11	L	75-117	L	0.26
		1,1,2-Trichloroethane	11	L	72-128	L	0.25
		1,3-Dichloropropane	12	L	77-118	L	0.22
		Dibromochloromethane	15	L	74-119	L	0.22
		1,2-Dibromoethane	18	L	77-123	L	0.15
		Tetrachloroethene	14	L	80-122	L	0.26
		1,1,1,2-Tetrachloroethane	12	L	73-128	L	0.22
		Bromoform	18	L	75-117	L	0.24
		1,1,2,2-Tetrachloroethane	13	L	79-111	L	0.35

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SW = surface water, EFF = effluents

L = Low range of the analytical curve

TABLE 5-20

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/kg
8021B	SED,S,HW	Dichlorodifluoromethane	13	L	44-125	L	0.96
		Chloromethane	13	L	61-140	L	1.00
		Vinyl chloride	10	L	73-120	L	0.70
		Bromomethane	11	L	60-124	L	0.39
		Chloroethane	11	L	62-127	L	0.89
		Trichlorofluoromethane	10	L	75-122	L	1.06
		1,1-Dichloroethene	11	L	85-120	L	1.02
		Methylene chloride	12	L	86-124	L	0.92
		t-1,2-Dichloroethene	15	L	80-121	L	0.62
		1,1-Dichloroethane	18	L	83-120	L	0.51
		c-1,2-Dichloroethene	15	L	90-121	L	0.56
		Bromochloromethane	10	L	80-118	L	0.47
		Chloroform	11	L	89-129	L	0.72
		2,2-Dichloropropane	19	L	88-121	L	0.77
		1,2-Dichloroethane	17	L	90-120	L	0.56
		1,1,1-Trichloroethane	16	L	91-123	L	0.67
		1,1-Dichloropropene	19	L	87-120	L	0.62
		Carbon tetrachloride	16	L	91-118	L	0.49
		Dibromomethane	10	L	68-118	L	0.47
		1,2-Dichloropropane	18	L	97-124	L	0.50
		Trichloroethene	18	L	88-118	L	0.72
		Bromodichloromethane	18	L	80-132	L	0.49
		c-1,3-Dichloropropene	12	L	89-119	L	0.54
		t-1,3-Dichloropropene	11	L	90-113	L	0.59
		1,1,2-Trichloroethane	11	L	77-126	L	0.71
		1,3-Dichloropropane	12	L	83-129	L	0.59
		Dibromochloromethane	15	L	87-119	L	0.57
		1,2-Dibromoethane	18	L	79-122	L	0.49
		Tetrachloroethene	16	L	94-117	L	0.60
		1,1,1,2-Tetrachloroethane	12	L	85-126	L	0.44
		Bromoform	18	L	79-123	L	0.54
		1,1,2,2-Tetrachloroethane	13	L	74-126	L	0.76
		Benzene	18	L	81-114	L	0.24
		Toluene	18	L	81-116	L	0.26
		Chlorobenzene	15	L	89-118	L	0.47
		Ethylbenzene	19	L	81-119	L	0.22
		m,p-Xylene	18	L	83-121	L	0.44
		Styrene	19	L	85-113	L	0.28
		o-Xylene	18	L	83-120	L	0.15
		Isopropylbenzene	18	L	82-114	L	0.31
		Bromobenzene	17	L	78-113	L	0.28

TABLE 5-20 cont

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/kg
8021B	SED,S,HW	n-Propylbenzene	19	L	88-111	L	0.15
		2-Chlorotoluene	19	L	85-116	L	0.47
		4-Chlorotoluene	18	L	85-122	L	0.43
		1,3,5-Trimethylbenzene	18	L	81-126	L	0.24
		t-Butylbenzene	18	L	77-130	L	0.24
		1,2,4-Trimethylbenzene	19	L	73-134	L	0.70
		s-Butylbenzene	17	L	73-140	L	0.40
		1,3-Dichlorobenzene	14	L	77-126	L	0.34
		p-Isopropyltoluene	12	L	72-118	L	0.40
		1,4-Dichlorobenzene	16	L	87-112	L	0.44
		1,2-Dichlorobenzene	18	L	80-118	L	0.48
		n-Butylbenzene	23	L	78-118	L	0.94
		1,2,4-Trichlorobenzene	23	L	65-119	L	1.09
		Naphthalene	27	L	60-138	L	0.70
		Hexachlorobutadiene	11	L	60-142	L	1.05
		1,2,3-Trichlorobenzene	24	L	53-142	L	0.77
		1,2-Dibromo-3-chloropropane	18	L	60-133	L	1.07
		1,2,3-Trichloropropane	19	L	66-131	L	0.64
		Methyl-t-butylether	20	L	60-139	L	3.90
		Isopropyl Ether	19	L	76-136	L	3.80

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SED = sediments, S= soil, HW = hazardous waste

L = Low range of the analytical curve

TABLE 5-21

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
8021B	GW,SW	Dichlorodifluoromethane	13	L	61-109	L	0.47
		Chloromethane	13	L	67-116	L	0.92
		Vinyl chloride	10	L	74-120	L	0.47
		Bromomethane	11	L	72-109	L	0.81
		Chloroethane	11	L	77-114	L	0.91
		Trichlorofluoromethane	10	L	72-122	L	0.67
		1,1-Dichloroethene	11	L	81-119	L	0.59
		Methylene chloride	12	L	73-128	L	0.35
		t-1,2-Dichloroethene	15	L	83-115	L	0.35
		1,1-Dichloroethane	18	L	74-121	L	0.24
		c-1,2-Dichloroethene	15	L	82-116	L	0.25
		Bromochloromethane	10	L	73-118	L	0.24
		Chloroform	11	L	81-121	L	0.28
		2,2-Dichloropropane	19	L	87-116	L	0.44
		1,2-Dichloroethane	17	L	73-130	L	0.24
		1,1,1-Trichloroethane	16	L	79-116	L	0.34
		1,1-Dichloropropene	19	L	88-108	L	0.18
		Carbon tetrachloride	16	L	77-118	L	0.24
		Dibromomethane	10	L	77-114	L	0.22
		1,2-Dichloropropane	18	L	79-121	L	0.17
		Trichloroethene	18	L	79-123	L	0.25
		Bromodichloromethane	18	L	73-128	L	0.17
		c-1,3-Dichloropropene	12	L	74-117	L	0.22
		t-1,3-Dichloropropene	11	L	75-117	L	0.26
		1,1,2-Trichloroethane	11	L	72-128	L	0.25
		1,3-Dichloropropane	12	L	77-118	L	0.22
		Dibromochloromethane	15	L	74-119	L	0.22
		1,2-Dibromoethane	18	L	77-123	L	0.15
		Tetrachloroethene	16	L	80-122	L	0.26
		1,1,1,2-Tetrachloroethane	12	L	73-128	L	0.22
		Bromoform	18	L	75-117	L	0.24
		1,1,2,2-Tetrachloroethane	13	L	79-111	L	0.35
		Benzene	17	L	84-118	L	0.17
		Toluene	17	L	80-121	L	0.24
		Chlorobenzene	15	L	82-125	L	0.24
		Ethylbenzene	17	L	80-125	L	0.15
		m,p-Xylene	17	L	78-132	L	0.48
		Styrene	19	L	68-130	L	0.25
		o-Xylene	17	L	81-127	L	0.25
		Isopropylbenzene	18	L	75-120	L	0.25
		Bromobenzene	17	L	76-122	L	0.17

TABLE 5-21 cont

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
8021B	GW,SW	n-Propylbenzene	19	L	80-120	L	0.31
		2-Chlorotoluene	19	L	72-118	L	0.28
		4-Chlorotoluene	18	L	64-136	L	0.24
		1,3,5-Trimethylbenzene	18	L	70-122	L	0.51
		t-Butylbenzene	18	L	79-121	L	0.35
		1,2,4-Trimethylbenzene	19	L	81-126	L	0.35
		s-Butylbenzene	17	L	79-123	L	0.26
		1,3-Dichlorobenzene	14	L	80-121	L	0.35
		p-Isopropyltoluene	12	L	76-113	L	0.36
		1,4-Dichlorobenzene	16	L	84-115	L	0.18
		1,2-Dichlorobenzene	18	L	76-120	L	0.38
		n-Butylbenzene	23	L	71-116	L	0.51
		1,2,4-Trichlorobenzene	23	L	74-121	L	0.59
		Naphthalene	27	L	72-131	L	0.94
		Hexachlorobutadiene	11	L	75-112	L	0.94
		1,2,3-Trichlorobenzene	24	L	75-115	L	0.46
		1,2-Dibromo-3-chloropropane	18	L	63-117	L	0.72
		1,2,3-Trichloropropane	19	L	74-111	L	0.35
		Methyl-t-butylether	16	L	68-124	L	1.16
		Isopropyl Ether	19	L	77-130	L	0.64

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

GW = ground water, SW = surface water

L = Low range of the analytical curve

TABLE 5-22

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
624	SW,EFF	Dichlorodifluoromethane	28	L	45-141	L	0.50
		Chloromethane	25	L	41-154	L	2.90
		Vinyl chloride	29	L	24-150	L	0.40
		Bromomethane	58	L	20-143	L	1.70
		Chloroethane	20	L	36-140	L	2.20
		Trichlorofluoromethane	17	L	63-114	L	2.40
		1,1-Dichloroethene	16	L	58-138	L	1.70
		Methylene chloride	19	L	40-130	L	0.36
		Carbon disulfide	25	L	62-136	L	0.70
		t-1,2-Dichloroethene	17	L	48-110	L	0.50
		1,1-Dichloroethane	16	L	44-115	L	0.21
		c-1,2-Dichloroethene	20	L	42-118	L	0.50
		Chloroform	16	L	59-109	L	0.90
		1,2-Dichloroethane	21	L	30-130	L	0.80
		1,1,1-Trichloroethane	15	L	64-112	L	0.50
		Carbon tetrachloride	22	L	33-147	L	2.00
		Benzene	15	L	58-135	L	0.30
		1,2-Dichloropropane	30	L	72-110	L	0.50
		Trichloroethene	22	L	52-143	L	0.50
		Bromodichloromethane	34	L	82-110	L	0.50
		c-1,3-Dichloropropene	29	L	70-125	L	0.30
		t-1,3-Dichloropropene	28	L	61-126	L	0.60
		1,1,2-Trichloroethane	28	L	72-127	L	1.90
		Toluene	20	L	56-135	L	0.70
		Dibromochloromethane	29	L	59-109	L	0.50
		Tetrachloroethene	32	L	68-128	L	0.50
		1,1,1,2-Tetrachloroethane	30	L	79-113	L	0.90
		Chlorobenzene	19	L	56-126	L	0.60
		Ethylbenzene	32	L	62-124	L	0.70
		m,p-Xylene	33	L	67-125	L	0.80
		Bromoform	30	L	51-114	L	1.20
		Styrene	39	L	62-106	L	0.50
		o-Xylene	33	L	64-128	L	0.60
		1,1,2,2-Tetrachloroethene	22	L	33-127	L	0.60
		Acrolein	27	L	65-133	L	0.97
		Acrylonitrile	26	L	62-128	L	0.95

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SW = surface water, EFF = effluents

L = Low range of the analytical curve

qa5tb4o

TABLE 5-23

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/kg
8260B	SED,S,HW	Dichlorodifluoromethane	30	L	40-130	L	0.90
		Chloromethane	16	L	41-154	L	1.00
		Vinyl chloride	32	L	20-150	L	0.50
		Bromomethane	31	L	19-143	L	0.60
		Chloroethane	29	L	36-140	L	0.80
		Trichlorofluoromethane	16	L	63-114	L	1.20
		Acetone	50	L	32-122	L	4.40
		1,1-Dichloroethene	19	L	58-138	L	0.40
		Methylene chloride	19	L	40-130	L	6.00
		Carbon disulfide	17	L	40-146	L	0.70
		t-1,2-Dichloroethene	14	L	45-139	L	0.50
		MTBE	18	L	54-128	L	0.90
		1,1-Dichloroethane	20	L	44-148	L	0.50
		2-Butanone	49	L	23-140	L	0.50
		IPE	35	L	49-151	L	0.60
		c-1,2-Dichloroethene	19	L	42-135	L	0.90
		Bromochloromethane	19	L	50-156	L	0.70
		Chloroform	16	L	54-145	L	0.50
		2,2-Dichloropropane	15	L	48-141	L	0.60
		1,2-Dichloroethane	17	L	30-135	L	0.60
		1,1,1-Trichloroethane	17	L	51-139	L	0.40
		1,1-Dichloropropene	17	L	52-138	L	1.00
		Carbon tetrachloride	24	L	55-135	L	0.40
		Benzene	17	L	58-135	L	0.70
		Dibromomethane	18	L	52-146	L	1.00
		1,2-Dichloropropane	22	L	57-146	L	0.90
		Trichloroethene	18	L	52-143	L	0.50
		Bromodichloromethane	19	L	64-135	L	1.00
		c-1,3-Dichloropropene	26	L	54-136	L	0.80
		MIBK	34	L	34-143	L	0.80
		t-1,3-Dichloropropene	19	L	51-137	L	0.50
		1,1,2-Trichloroethane	22	L	57-137	L	0.90
		Toluene	18	L	56-135	L	0.70
		1,3-Dichloropropane	28	L	54-150	L	1.00
		2-Hexanone	30	L	17-155	L	1.30
		Dibromochloromethane	23	L	49-137	L	0.50
		1,2-Dibromoethane	18	L	46-138	L	1.60
		Tetrachloroethene	20	L	58-136	L	0.50
		1,1,1,2-Tetrachloroethane	25	L	52-141	L	0.80
		Chlorobenzene	14	L	54-136	L	0.80
		Ethylbenzene	26	L	55-133	L	1.00

TABLE 5-23 cont

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/kg
8260B	SED,S,HW	m,p-Xylene	23	L	56-137	L	1.20
		Bromoform	24	L	40-133	L	0.70
		Styrene	23	L	47-137	L	0.70
		o-Xylene	20	L	55-139	L	0.70
		1,1,2,2-Tetrachloroethene	23	L	33-135	L	1.00
		1,2,3-Trichloropropane	29	L	31-138	L	1.20
		Isopropylbenzene	18	L	57-141	L	0.80
		Bromobenzene	18	L	52-144	L	0.80
		Propylbenzene	17	L	57-134	L	0.90
		2-Chlorotoluene	18	L	56-137	L	1.00
		4-Chlorotoluene	16	L	51-138	L	0.70
		1,3,5-Trimethylbenzene	18	L	58-139	L	0.60
		t-Butylbenzene	18	L	58-141	L	0.80
		1,2,4-Trimethylbenzene	20	L	54-146	L	0.80
		sec-Butylbenzene	20	L	55-142	L	0.90
		1,3-Dichlorobenzene	22	L	57-134	L	0.70
		1,4-Dichlorobenzene	20	L	55-137	L	0.80
		1,2Dichlorobenzene	25	L	57-141	L	0.70
		4-Isopropyltoluene	19	L	51-146	L	0.60
		Butylbenzene	17	L	50-135	L	0.60
		1,2,4-Trichlorobenzene	37	L	37-146	L	1.00
		Naphthalene	20	L	27-153	L	0.90
		Hexachlorobutadiene	39	L	49-130	L	0.60
		1,2,3-Trichlorobenzene	37	L	36-130	L	1.00

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SED = sediments, S = soil, HW = hazardous waste

L = Low range of the analytical curve

TABLE 5-24

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
8260B	SW,GW	Dichlorodifluoromethane	28	L	45-141	L	0.50
		Chloromethane	25	L	41-154	L	2.90
		Vinyl chloride	29	L	24-150	L	0.40
		Bromomethane	58	L	20-143	L	1.70
		Chloroethane	20	L	36-140	L	2.20
		Trichlorofluoromethane	17	L	63-114	L	2.40
		Acetone	32	L	32-122	L	6.85
		1,1-Dichloroethene	16	L	58-138	L	1.70
		Methylene chloride	19	L	40-130	L	0.36
		Carbon disulfide	25	L	62-136	L	0.70
		t-1,2-Dichloroethene	17	L	48-110	L	0.50
		MTBE	26	L	54-128	L	0.50
		1,1-Dichloroethane	16	L	44-115	L	050
		2-Butanone	59	L	23-140	L	3.00
		IPE	28	L	50-140	L	0.30
		c-1,2-Dichloroethene	20	L	42-118	L	0.50
		Bromochloromethane	21	L	65-114	L	0.70
		Chloroform	16	L	59-109	L	0.90
		2,2-Dichloropropane	19	L	50-119	L	1.40
		1,2-Dichloroethane	21	L	30-130	L	0.80
		1,1,1-Trichloroethane	15	L	64-112	L	0.50
		1,1-Dichloropropene	16	L	59-111	L	0.50
		Carbon tetrachloride	22	L	33-147	L	2.00
		Benzene	15	L	58-135	L	0.30
		Dibromomethane	26	L	77-121	L	0.50
		1,2-Dichloropropane	30	L	72-110	L	0.50
		Trichloroethene	22	L	52-143	L	0.50
		Bromodichloromethane	34	L	82-110	L	0.50
		c-1,3-Dichloropropene	29	L	70-125	L	0.30
		MIBK	26	L	34-143	L	1.00
		t-1,3-Dichloropropene	28	L	61-126	L	0.60
		1,1,2-Trichloroethane	28	L	72-127	L	1.90
		Toluene	20	L	56-135	L	0.70
		1,3-Dichloropropane	28	L	61-130	L	0.50
		2-Hexanone	27	L	17-155	L	1.90
		Dibromochloromethane	29	L	59-109	L	0.50
		1,2-Dibromoethane	24	L	46-116	L	0.50
		Tetrachloroethene	32	L	68-128	L	0.50
		1,1,1,2-Tetrachloroethane	30	L	79-103	L	0.90
		Chlorobenzene	19	L	56-126	L	0.60
		Ethylbenzene	32	L	62-124	L	0.70

TABLE 5-24 cont

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
8260B	SW,GW	m,p-Xylene	33	L	67-125	L	0.80
		Bromoform	30	L	51-114	L	1.20
		Styrene	39	L	62-106	L	0.50
		o-Xylene	33	L	64-128	L	0.60
		1,1,2,2-Tetrachloroethene	22	L	33-127	L	0.60
		1,2,3-Trichloropropane	24	L	19-130	L	0.50
		Isopropylbenzene	34	L	72-111	L	0.40
		Bromobenzene	31	L	56-117	L	0.50
		Propylbenzene	35	L	65-112	L	0.40
		2-Chlorotoluene	33	L	71-109	L	0.60
		4-Chlorotoluene	32	L	61-110	L	0.50
		1,3,5-Trimethylbenzene	36	L	68-109	L	0.60
		t-Butylbenzene	33	L	70-110	L	0.50
		1,2,4-Trimethylbenzene	36	L	68-116	L	0.80
		sec-Butylbenzene	33	L	68-118	L	0.50
		1,3-Dichlorobenzene	32	L	77-112	L	0.30
		1,4-Dichlorobenzene	32	L	75-123	L	0.40
		1,2Dichlorobenzene	28	L	72-124	L	0.40
		4-Isopropyltoluene	36	L	70-115	L	0.40
		Butylbenzene	34	L	60-119	L	0.40
		1,2,4-Trichlorobenzene	41	L	53-111	L	0.60
		Naphthalene	43	L	27-149	L	1.70
		Hexachlorobutadiene	44	L	60-129	L	1.40
		1,2,3-Trichlorobenzene	46	L	43-129	L	0.50

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SW = surface water, GW = ground water

L = Low range of the analytical curve

TABLE 5-25

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
625	SW,EFF	Acenaphthene	20	L	49-119	L	1.05
		Acenaphthylene	36	L	52-125	L	1.03
		Aniline	19	L	39-126	L	3.23
		Anthracene	14	L	74-103	L	1.21
		Benzidine	38	L	10-142	L	7.11
		Benzoic Acid	52	L	49-139	L	1.04
		Benzo(a)anthracene	21	L	71-116	L	1.03
		Benzo(a)pyrene	16	L	75-118	L	0.91
		Benzo(b)fluoranthene	26	L	56-125	L	0.83
		Benzo(ghi)perylene	48	L	24-145	L	2.54
		Benzo(k)fluoranthene	30	L	57-145	L	0.83
		4-Bromophenylphenyl ether	37	L	44-120	L	0.85
		Butylbenzylphthalate	40	L	52-131	L	6.74
		Carbazole	22	L	64-110	L	1.29
		4-Chloro-3-methylphenol	19	L	50-124	L	1.56
		4-Chloroaniline	24	L	56-115	L	1.43
		Bis(2-chloroethoxy)methane	30	L	52-112	L	1.72
		Bis(2-chloroethyl)ether	23	L	55-110	L	1.94
		Bis(2-chloroisopropyl)ether	40	L	23-113	L	1.41
		2-Chloronaphthalene	44	L	38-127	L	0.90
		2-Chlorophenol	19	L	48-121	L	2.43
		4-Chlorophenylphenyl ether	35	L	57-128	L	0.93
		Chrysene	16	L	67-142	L	1.03
		Dibenzofuran	32	L	58-122	L	0.98
		Dibenz(ah)anthracene	38	L	49-140	L	2.28
		1,2-Dichlorobenzene	38	L	36-114	L	1.34
		1,3-Dichlorobenzene	41	L	19-100	L	1.21
		1,4-Dichlorobenzene	21	L	23-116	L	1.89
		3,3'-Dichlorobenzidine	56	L	D-127	L	3.97
		2,4-Dichlorophenol	39	L	45-124	L	1.33
		Diethylphthalate	23	L	75-135	L	1.48
		2,4-Dimethylphenol	32	L	40-106	L	2.68
		Dimethylphthalate	50	L	52-127	L	1.36
		Di-n-butylphthalate	50	L	35-136	L	3.00
		4,6-Dinitro-2-methylphenol	60	L	27-173	L	1.64
		2,4-Dinitrophenol	60	L	20-138	L	2.37
		2,4-Dinitrotoluene	18	L	46-147	L	1.29
		2,6-Dinitrotoluene	45	L	43-150	L	1.30
		Di-n-octylphthalate	40	L	59-139	L	4.67
		1,2-Diphenylhydrazine	19	L	64-130	L	1.51
		Fluoranthene	15	L	62-117	L	0.92

TABLE 5-25 cont

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
625	SW,EFF	Hexachlorobenzene	49	L	49-137	L	0.86
		Flrorene	31	L	51-119	L	0.80
		Hexachlorobutadiene	42	L	38-133	L	2.01
		Hexachlorocyclopentadiene	42	L	28-137	L	2.52
		Hexachloroethane	38	L	29-130	L	1.34
		Indeno(123-cd)pyrene	49	L	26-143	L	4.58
		Isophorone	28	L	58-126	L	1.70
		2-Methylnaphthalene	5	L	55-112	L	0.98
		2-Methylphenol	19	L	41-117	L	3.50
		4-methylphenol	30	L	31-115	L	1.80
		2-Nitroaniline	33	L	58-123	L	2.55
		4-Nitroaniline	32	L	46-124	L	2.22
		Nitrobenzene	26	L	49-104	L	0.94
		2-Nitrophenol	39	L	41-118	L	1.60
		4-Nitrophenol	48	L	24-127	L	3.88
		N-nitroso-di-n-propylamine	18	L	38-148	L	2.22
		N-nitroso-di-phenylamine	35	L	27-127	L	2.82
		N-nitrosodimethylamine	19	L	32-124	L	2.18
		Pentachlorophenol	20	L	26-137	L	1.17
		Phenanthrene	19	L	73-111	L	1.21
		Phenol	20	L	22-116	L	4.33
		Pyrene	18	L	32-132	L	1.27
		Bis(2-ethylhexyl)phthalate	23	L	79-146	L	1.72
		1,2,4-Trichlorobenzene	20	L	27-121	L	1.78
		2,4,5-Trichlorophenol	32	L	51-130	L	1.76
		2,4,6-Trichlorophenol	40	L	46-134	L	1.42
		3-Nitroaniline	19	L	62-114	L	1.67
		Naphthalene	32	L	41-106	L	0.79

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SW = surface water, EFF = effluents

L = Low range of the analytical curve

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TABLE 5-26

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
8270C	GW,SW	Acenaphthene	20	L	49-119	L	1.05
		Acenaphthylene	36	L	52-125	L	1.03
		Aniline	19	L	39-126	L	3.23
		Anthracene	14	L	74-103	L	1.21
		Benzidine	38	L	10-142	L	7.11
		Benzoic Acid	52	L	49-139	L	1.04
		Benzo(a)anthracene	21	L	71-116	L	1.03
		Benzo(a)pyrene	16	L	75-118	L	0.91
		Benzo(b)fluoranthene	26	L	56-125	L	0.83
		Benzo(ghi)perylene	48	L	24-145	L	2.54
		Benzo(k)fluoranthene	30	L	57-145	L	0.83
		4-Bromophenylphenyl ether	37	L	44-120	L	0.85
		Butylbenzylphthalate	40	L	52-131	L	1.74
		Carbazole	22	L	64-110	L	1.29
		4-Chloro-3-methylphenol	19	L	50-124	L	1.56
		4-Chloroaniline	24	L	56-115	L	1.43
		Bis(2-chloroethoxy)methane	30	L	52-112	L	1.72
		Bis(2-chloroethyl)ether	23	L	56-110	L	1.94
		Bis(2-chloroisopropyl)ether	40	L	23-113	L	1.41
		2-Chloronaphthalene	44	L	38-127	L	0.90
		2-Chlorophenol	19	L	48-121	L	2.43
		4-Chlorophenylphenyl ether	35	L	57-128	L	0.93
		Chrysene	16	L	67-142	L	1.03
		Dibenzofuran	32	L	58-122	L	0.98
		Dibenz(ah)anthracene	38	L	49-140	L	2.28
		1,2-Dichlorobenzene	38	L	36-114	L	1.34
		1,3-Dichlorobenzene	41	L	19-100	L	1.21
		1,4-Dichlorobenzene	21	L	23-116	L	1.89
		3,3'-Dichlorobenzidine	56	L	D-127	L	3.97
		2,4-Dichlorophenol	39	L	45-124	L	1.33
		Diethylphthalate	23	L	75-135	L	1.48
		2,4-Dimethylphenol	32	L	40-106	L	2.68
		Dimethylphthalate	50	L	52-127	L	1.36
		Di-n-butylphthalate	50	L	35-136	L	3.00
		4,6-Dinitro-2-methylphenol	60	L	27-173	L	1.64
		2,4-Dinitrophenol	60	L	20-138	L	2.37
		2,4-Dinitrotoluene	18	L	46-147	L	1.29
		2,6-Dinitrotoluene	45	L	43-150	L	1.30
		Di-n-octylphthalate	40	L	59-139	L	4.67
		1,2-Diphenylhydrazine	19	L	64-130	L	1.51
		Fluoranthene	15	L	62-117	L	0.92

TABLE 5-26 cont

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
8270C	GW,SW	Fluorene	31	L	51-119	L	0.80
		Hexachlorobenzene	49	L	49-137	L	0.86
		Hexachlorobutadiene	42	L	38-133	L	2.01
		Hexachlorocyclopentadiene	42	L	28-137	L	2.52
		Hexachloroethane	38	L	29-130	L	1.34
		Indeno(123-cd)pyrene	49	L	26-143	L	4.58
		Isophorone	28	L	58-126	L	1.70
		2-Methylnaphthalene	25	L	55-112	L	0.98
		2-Methylphenol	19	L	41-117	L	3.50
		4-methylphenol	30	L	31-115	L	1.80
		2-Nitroaniline	33	L	58-123	L	2.55
		4-Nitroaniline	32	L	46-124	L	2.22
		Nitrobenzene	26	L	49-104	L	0.94
		2-Nitrophenol	39	L	41-118	L	1.60
		4-Nitrophenol	48	L	24-127	L	3.88
		N-nitroso-di-n-propylamine	18	L	38-148	L	2.22
		N-nitroso-di-phenylamine	35	L	27-127	L	2.82
		N-nitrosodimethylamine	19	L	32-124	L	2.18
		Pentachlorophenol	20	L	26-137	L	1.17
		Phenanthrene	19	L	73-111	L	1.21
		Phenol	20	L	22-116	L	4.33
		Pyrene	18	L	32-132	L	1.27
		Bis(2-ethylhexyl)phthalate	23	L	79-146	L	4.72
		1,2,4-Trichlorobenzene	20	L	27-121	L	1.78
		2,4,5-Trichlorophenol	32	L	51-130	L	3.79
		2,4,6-Trichlorophenol	40	L	46-134	L	1.42
		3-Nitroaniline	19	L	62-114	L	1.67
		Naphthalene	32	L	41-106	L	0.79
		Pyridine	28	L	35-127	L	2.85

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

GW = ground water, SW = surface water

L = Low range of the analytical curve

qa5tb4s.

TABLE 5-27

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL mg/kg
8270C	SED,S,HW	Acenaphthene	24	L	35-122	L	0.07
		Acenaphthylene	20	L	27-125	L	0.07
		Aniline	52	L	29-118	L	0.06
		Anthracene	17	L	36-160	L	0.04
		Benzoic Acid	88	L	7-168	L	0.06
		Benzo(a)anthracene	21	L	37-149	L	0.04
		Benzo(a)pyrene	20	L	31-157	L	0.03
		Benzo(b)fluoranthene	26	L	24-144	L	0.03
		Benzo(ghi)perylene	46	L	16-142	L	0.07
		Benzo(k)fluoranthene	43	L	34-160	L	0.05
		4-Bromophenylphenyl ether	33	L	35-138	L	0.05
		Butylbenzylphthalate	17	L	58-140	L	0.04
		Carbazole	18	L	33-149	L	0.03
		4-Chloro-3-methylphenol	24	L	24-117	L	0.08
		4-Chloroaniline	57	L	28-128	L	0.06
		Bis(2-chloroethoxy)methane	19	L	36-145	L	0.08
		Bis(2-chloroethyl)ether	21	L	34-159	L	0.08
		Bis(2-chloroisopropyl)ether	32	L	27-140	L	0.05
		2-Chloronaphthalene	29	L	29-150	L	0.08
		2-Chlorophenol	33	L	18-120	L	0.08
		4-Chlorophenylphenyl ether	19	L	32-148	L	0.08
		Chrysene	11	L	76-140	L	0.04
		Dibenzofuran	17	L	37-147	L	0.08
		Dibenz(ah)anthracene	70	L	26-143	L	0.07
		1,2-Dichlorobenzene	31	L	43-157	L	0.10
		1,3-Dichlorobenzene	25	L	40-147	L	0.09
		1,4-Dichlorobenzene	26	L	25-118	L	0.09
		2,4-Dichlorophenol	25	L	28-116	L	0.08
		Diethylphthalate	18	L	40-150	L	0.08
		2,4-Dimethylphenol	23	L	28-117	L	0.07
		Dimethylphthalate	23	L	38-143	L	0.07
		Di-n-butylphthalate	11	L	33-145	L	0.10
		4,6-Dinitro-2-methylphenol	62	L	25-142	L	0.09
		2,4-Dinitrophenol	51	L	16-132	L	0.07
		2,4-Dinitrotoluene	27	L	21-118	L	0.05
		2,6-Dinitrotoluene	42	L	37-140	L	0.06
		Di-n-octylphthalate	43	L	25-150	L	0.04
		1,2-Diphenylhydrazine	24	L	36-143	L	0.10
		Fluoranthene	15	L	34-153	L	0.03
		Fluorene	16	L	34-162	L	0.09

TABLE 5-27 cont

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL mg/kg
8270C	SED,S,HW	Hexachlorobenzene	35	L	32-140	L	0.05
		Hexachlorobutadiene	33	L	37-141	L	0.10
		Hexachlorocyclopentadiene	52	L	12-100	L	0.10
		Hexachloroethane	23	L	39-160	L	0.10
		Indeno(123-cd)pyrene	48	L	24-135	L	0.06
		Isophorone	19	L	35-151	L	0.09
		2-Methylnaphthalene	14	L	20-140	L	0.15
		2-Methylphenol	27	L	35-152	L	0.09
		4-methylphenol	32	L	42-151	L	0.09
		2-Nitroaniline	29	L	36-140	L	0.06
		4-Nitroaniline	37	L	34-142	L	0.04
		Nitrobenzene	18	L	36-149	L	0.08
		2-Nitrophenol	35	L	35-131	L	0.07
		4-Nitrophenol	41	L	30-136	L	0.04
		N-nitroso-di-n-propylamine	35	L	29-133	L	0.09
		N-nitroso-di-phenylamine	24	L	33-151	L	0.04
		N-nitrosodimethylamine	28	L	39-148	L	0.06
		Pentachlorophenol	45	L	25-126	L	0.04
		Phenanthrene	17	L	35-140	L	0.05
		Phenol	27	L	23-125	L	0.09
		Pyrene	19	L	32-124	L	0.04
		Bis(2-ethylhexyl)phthalate	19	L	30-150	L	0.05
		1,2,4-Trichlorobenzene	38	L	30-114	L	0.08
		2,4,5-Trichlorophenol	22	L	23-140	L	0.08
		2,4,6-Trichlorophenol	37	L	20-145	L	0.08
		3-Nitroaniline	25	L	35-141	L	0.04
		Naphthalene	15	L	40-152	L	0.07

Note: MDL = Method detection limit as determined by 40CFR136, Appendix B Rev 1.11

SED = sediments, S = soil, HW = hazardous waste

L = Low range of the analytical curve

TABLE 5-28

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
608	SW,EFF	a-BHC	25	L	57-107	L	0.0008
		Lindane	22	L	60-121	L	0.0009
		b-BHC	26	L	74-126	L	0.0006
		d-BHC	29	L	18-128	L	0.0006
		Aldrin	25	L	67-129	L	0.0007
		4,4'-DDE	23	L	79-125	L	0.0007
		4,4'-DDT	14	L	64-129	L	0.0003
		4,4'-DDD	24	L	67-137	L	0.0008
		Heptachlor	19	L	71-124	L	0.0009
		Heptachlor epoxide	25	L	78-130	L	0.0009
		Endosulfan I	23	L	73-119	L	0.0015
		Dieldrin	26	L	76-128	L	0.0005
		Endrin	20	L	54-138	L	0.0003
		Endosulfan II	25	L	57-127	L	0.0009
		Endrin Aldehyde	27	L	61-139	L	0.0009
		Endosulfan Sulfate	28	L	40-116	L	0.0004
		Toxaphene	11	L	33-112	L	0.0287
		g-Chlordane	22	L	75-121	L	0.0008
		a-Chlordane	23	L	73-119	L	0.0010
		Endrin Ketone	30	L	56-136	L	0.0005
		Methoxychlor	22	L	84-128	L	0.0011
		PCB 1016	15	L	78-164	L	0.0117
		PCB 1221	31	L	57-133	L	0.0394
		PCB1232	30	L	26-135	L	0.0142
		PCB1242	26	L	62-144	L	0.0208
		PCB1248	16	L	56-150	L	0.0265
		PCB1254	14	L	66-140	L	0.0212
		PCB1260	26	L	60-141	L	0.0254

Note: MDL = Method detection limit as determined by 40CFR136, Appendix B Rev 1.11

SW = surface water, EFF = effluents

L = Low range of the analytical curve

TABLE 5-29

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
8081A	GW,SW, HW	a-BHC	25	L	57-107	L	0.0008
PEST		Lindane	22	L	60-121	L	0.0009
		b-BHC	26	L	74-126	L	0.0006
8082		d-BHC	29	L	18-128	L	0.0006
PCB		Aldrin	25	L	67-129	L	0.0007
		4,4'-DDE	23	L	79-125	L	0.0007
		4,4'-DDT	14	L	64-129	L	0.0003
		4,4'-DDD	24	L	67-137	L	0.0008
		Heptachlor	19	L	71-124	L	0.0009
		Heptachlor epoxide	25	L	78-130	L	0.0009
		Endosulfan I	23	L	73-119	L	0.0015
		Dieldrin	26	L	76-128	L	0.0005
		Endrin	20	L	54-138	L	0.0003
		Endosulfan II	25	L	57-127	L	0.0009
		Endrin Aldehyde	27	L	61-139	L	0.0009
		Endosulfan Sulfate	28	L	40-116	L	0.0004
		Methoxychlor	22	L	84-128	L	0.0011
		Toxaphene	11	L	33-112	L	0.0287
		g-Chlordane	22	L	75-121	L	0.0008
		a-Chlordane	23	L	73-119	L	0.0010
		Endrin Ketone	30	L	56-136	L	0.0005
		PCB1016	15	L	78-164	L	0.0117
		PCB1221	31	L	57-133	L	0.0394
		PCB1232	30	L	26-135	L	0.0142
		PCB1242	26	L	62-144	L	0.0208
		PCB1248	16	L	56-150	L	0.0265
		PCB1254	14	L	66-140	L	0.0212
		PCB1260	26	L	60-141	L	0.0254

Note: MDL = Method detection limit as determined by 40CFR¶36, Appendix B Rev 1.11

SW = surface water, GW = ground water, HW = hazardous waste

L = Low range of the analytical curve

TABLE 5-30

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/kg
8081A	SED,S, HW	a-BHC	27	L	58-94	L	0.226
		Lindane	63	L	67-109	L	0.218
8082		b-BHC	30	L	74-134	L	0.234
PCB		d-BHC	33	L	67-113	L	1.199
		Aldrin	69	L	77-123	L	0.940
		4,4'-DDE	30	L	62-114	L	0.178
		4,4'-DDT	65	L	76-124	L	0.424
		4,4'-DDD	23	L	77-123	L	0.388
		Heptachlor	59	L	79-125	L	0.338
		Heptachlor epoxide	32	L	71-113	L	0.945
		Endosulfan I	36	L	64-116	L	0.555
		Dieldrin	78	L	76-136	L	0.354
		Endrin	82	L	79-129	L	0.208
		Endosulfan II	35	L	65-135	L	0.233
		Endrin Aldehyde	43	L	63-149	L	0.806
		Endosulfan Sulfate	42	L	23-109	L	0.637
		Methoxychlor	31	L	69-131	L	0.347
		Toxaphene	24	L	32-126	L	38.2
		g-Chlordane	25	L	70-118	L	0.384
		a-Chlordane	31	L	69-111	L	0.260
		Endrin Ketone	50	L	42-142	L	0.372
		PCB1016	50	L	31-122	L	18.1
		PCB1221	40	L	37-146	L	22.6
		PCB1232	31	L	38-154	L	18.0
		PCB1242	28	L	26-171	L	29.1
		PCB1248	26	L	27-139	L	20.1
		PCB1254	33	L	28-143	L	13.0
		PCB1260	29	L	34-144	L	12.6

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SED = sediments, S = soil, HW = hazardous waste

L = Low range of the analytical curve

TABLE 5-31

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
8310	SW,GW	Naphthalene	15	L	51-122	L	0.30
HPLC		Acenaphthene	27	L	42-118	L	0.17
		Anthracene	19	L	25-112	L	0.06
		Fluoranthene	19	L	63-126	L	0.03
		Fluorene	24	L	57-114	L	0.09
		Pyrene	20	L	60-127	L	0.03
		Benzo(a)anthracene	16	L	62-112	L	0.01
		Benzo(a)pyrene	17	L	51-119	L	0.01
		Benzo(b)fluoranthene	16	L	58-128	L	0.02
		Benzo(k)fluroanthene	20	L	57-128	L	0.01
		Chrysene	17	L	56-125	L	0.01
		Dibenzo(a,h)anthracene	25	L	57-133	L	0.02
		Indeno(1,2,3-cd)pyrene	22	L	59-127	L	0.03
		Acenaphthylene	30	L	47-125	L	0.18
		Benzo(g,h,i)perylene	28	L	60-128	L	0.05
		Phenanthrene	19	L	51-125	L	0.09

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

GW = ground water, HW = hazardous waste, SW = surface water

L = Low range of the analytical curve

TABLE 5-32

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL mg/kg
8310 HPLC	SED,S,HW	Naphthalene	30	L	19-128	L	0.014
		Acenaphthene	50	L	43-130	L	0.026
		Anthracene	24	L	41-117	L	0.019
		Fluoranthene	20	L	44-130	L	0.004
		Fluorene	25	L	19-131	L	0.007
		Pyrene	27	L	41-132	L	0.006
		Benzo(a)anthracene	22	L	35-119	L	0.003
		Benzo(a)pyrene	24	L	12-125	L	0.005
		Benzo(b)fluoranthene	20	L	45-123	L	0.002
		Benzo(k)fluroanthene	22	L	42-122	L	0.002
		Chrysene	21	L	42-123	L	0.003
		Dibenzo(a,h)anthracene	27	L	27-145	L	0.004
		Indeno(1,2,3-cd)pyrene	27	L	42-126	L	0.006
		Acenaphthylene	30	L	43-138	L	0.031
		Benzo(g,h,i)perylene	28	L	36-138	L	0.005
		Phenanthrene	24	L	31-137	L	0.034

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SED = sediments, HW = hazardous waste, S = soil

L = Low range of the analytical curve

TABLE 5-33

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL mg/kg
8100 (GC)	SED,S,HW	Naphthalene	26	L	23-116	L	0.06
		Acenaphthene	25	L	28-119	L	0.05
		Anthracene	16	L	17-152	L	0.03
		Fluoranthene	18	L	41-124	L	0.03
		Fluorene	22	L	37-121	L	0.12
		Pyrene	22	L	44-125	L	0.03
		Benzo(a)anthracene	19	L	36-131	L	0.07
		Benzo(a)pyrene	22	L	34-124	L	0.01
		Benzo(b)fluoranthene	19	L	44-122	L	0.04
		Benzo(k)fluroanthene	20	L	51-128	L	0.04
		Chrysene	19	L	25-147	L	0.07
		Dibenzo(a,h)anthracene	26	L	18-134	L	0.02
		Indeno(1,2,3-cd)pyrene	26	L	17-134	L	0.06
		Acenaphthylene	25	L	28-118	L	0.05
		Benzo(g,h,i)perylene	22	L	16-135	L	0.02
		Phenanthrene	21	L	13-153	L	0.04
		1-Methylnaphthalene	19	L	32-114	L	0.15
		2-Methylnaphthalene	19	L	32-114	L	0.15

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SED = sediments, HW = hazardous waste, S = soil

L = Low range of the analytical curve

TABLE 5-34

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
8100	SW,GW	Naphthalene	22	L	58-117	L	1.72
(GC)		Acenaphthene	22	L	60-122	L	2.09
		Anthracene	13	L	52-132	L	1.15
		Fluorene	16	L	63-129	L	3.08
		Fluoranthene	14	L	69-126	L	0.84
		Pyrene	13	L	70-126	L	1.26
		Benzo(a)anthracene	13	L	57-125	L	1.05
		Benzo(a)pyrene	13	L	64-131	L	1.14
		Benzo(b)fluoranthene	14	L	59-133	L	1.01
		Benzo(k)fluroanthene	14	L	74-131	L	1.01
		Chrysene	16	L	45-149	L	1.30
		Dibenzo(a,h)anthracene	19	L	53-136	L	3.53
		Indeno(1,2,3-cd)pyrene	37	L	53-135	L	2.79
		Acenaphthylene	20	L	61-121	L	1.60
		Benzo(g,h,i)perylene	11	L	60-130	L	1.67
		Phenanthrene	16	L	58-130	L	1.30
		1-Methylnaphthalene	13	L	65-110	L	2.00
		2-Methylnaphthalene	12	L	65-110	L	1.95

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SW = surface water, GW = ground water

L = Low range of the analytical curve

TABLE 5-35

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
610	SW,EFF	Naphthalene	22	L	51-122	L	0.29
HPLC		Acenaphthene	22	L	58-123	L	0.17
		Anthracene	13	L	49-137	L	0.06
		Fluoranthene	14	L	66-125	L	0.03
		Fluorene	16	L	62-128	L	0.09
		Pyrene	13	L	61-129	L	0.03
		Benzo(a)anthracene	13	L	51-131	L	0.01
		Benzo(a)pyrene	13	L	61-128	L	0.01
		Benzo(b)fluoranthene	14	L	50-136	L	0.02
		Benzo(k)fluroanthene	14	L	63-136	L	0.01
		Chrysene	16	L	43-148	L	0.01
		Dibenzo(a,h)anthracene	19	L	45-135	L	0.02
		Indeno(1,2,3-cd)pyrene	37	L	39-139	L	0.03
		Acenaphthylene	20	L	57-122	L	0.18
		Benzo(g,h,i)perylene	11	L	40-140	L	0.05
		Phenanthrene	16	L	53-135	L	0.09

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SW = surface water, EFF = effluents

L = Low range of the analytical curve

TABLE 5-36

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
610 GC	SW,GW	Naphthalene	22	L	58-117	L	1.72
		Acenaphthene	22	L	60-122	L	2.09
		Anthracene	13	L	52-132	L	1.15
		Fluoranthene	14	L	69-126	L	0.84
		Fluorene	16	L	63-129	L	3.08
		Pyrene	13	L	70-126	L	1.26
		Benzo(a)anthracene	13	L	57-125	L	1.05
		Benzo(a)pyrene	13	L	64-131	L	1.14
		Benzo(b)fluoranthene	14	L	59-133	L	1.01
		Benzo(k)fluroanthene	14	L	74-131	L	1.01
		Chrysene	16	L	45-149	L	1.30
		Dibenzo(a,h)anthracene	19	L	53-136	L	3.53
		Indeno(1,2,3-cd)pyrene	37	L	53-135	L	2.79
		Acenaphthylene	20	L	61-121	L	1.60
		Benzo(g,h,i)perylene	11	L	60-130	L	1.67
		Phenanthrene	16	L	58-130	L	1.30
		1-Methylnaphthalene	13	L	65-110	L	2.00
		2-Methylnaphthalene	12	L	65-110	L	1.95

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SW = surface water, GW = ground water

L = Low range of the analytical curve

TABLE 5-37

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	CONC RANGE	ACCURACY %	CONC RANGE	MDL ug/l
8141A	SW,GW	Demeton	53	L	37-130	L	0.12
		Diazinon	36	L	68-132	L	0.2
		Disulfoton	37	L	70-121	L	0.1
		Methylparathion	43	L	32-112	L	0.1
		Ronnel	38	L	61-135	L	0.1
		Malathion	43	L	47-128	L	0.1
		Dursban	41	L	36-129	L	0.1
		Fenthion	39	L	18-130	L	0.1
		Parathion	46	L	69-115	L	0.1
		Guthion	46	L	37-142	L	0.5
8141A	SED,S,HW	Demeton	46	L	35-125	L	6
		Diazinon	39	L	50-125	L	10
		Disulfoton	41	L	62-131	L	5
		Methylparathion	38	L	40-129	L	5
		Ronnel	40	L	60-130	L	5
		Malathion	39	L	50-137	L	5
		Dursban	38	L	39-133	L	5
		Fenthion	39	L	20-122	L	5
		Parathion	44	L	61-124	L	5
		Guthion	40	L	35-149	L	25

Note: MDL = Method detection limit as determined by 40CFR 136, Appendix B Rev 1.11

SW = surface water, GW = ground water

L = Low range of the analytical curve

TABLE 5-38

QUALITY ASSURANCE OBJECTIVES

METHOD	MATRIX	ANALYTE	PRECISION %	ACCURACY %	MDL mg / m3
TO-3 EPA-18	Air, Bag	MTBE	1.50	93.3	0.10
		Benzene	1.29	95.5	0.05
		Toluene	1.41	96.0	0.06
		Ethyl Benzene	2.38	95.5	0.07
		m,p-Xylene	2.75	96.8	0.21
		o-Xylene	2.87	96.3	0.10
		C4 - C10	2.22	94.3	0.84

Note: Precision / Accuracy data from daily standard analyzed over seven days.

METHOD	MATRIX	ANALYTE	PRECISION %	ACCURACY %	DETECTION LIMIT mg
NIOSH GC	Charcoal Tube	Solvents	7	86-114	0.002
NIOSH GC	Passive Monitor	Solvents	9	82-118	0.002
NIOSH 7300 (ICP)	Filters	Metals	5	90-110	0.001

TABLE 5-39

SURROGATE RECOVERY RANGES

METHOD	MATRIX	COMPOUND	RANGE
8270C	SED,S,HW	Nitobenzene-D5	26-110
		2-Fluorobiphenyl	25-111
		Terphenyl-D14	35-132
		Phenol-D5	23-127
		2-Fluorophenol	23-117
		Triboromophenol	28-118
8270C	SW,GW	Nitrobenzene-D5	29-114
		2-Fluorobiphenyl	28-116
		Terphenyl-D14	10-121
		Phenol- D5	10-104
		2-Fluorophenol	10-80
		Tribromophenol	28-134
8310(HPLC)	SW,GW	p-Terphenyl-d14	22-116
8310(HPLC)	SED,S,HW	p-Terphenyl-d14	20-127
610 (HPLC)	SW,EFF	p-Terphenyl-d14	22-116
610(GC)	SW,EFF	2-Fluorobiphenyl	45-147
8100(GC)	SED,S,HW	2-Fluorobiphenyl	35-150
8100(GC)	SW,EFF	2-Fluorobiphenyl	45-147

TABLE 5-39 cont

SURROGATE RECOVERY RANGES

METHOD	MATRIX	COMPOUND	RANGE
8151A	SW,GW	Dichloroacetic acid	24-147
8081A	SW,GW	Tetrachloro-m-xylene	19-147
		Dibutylchlorendate	10-135
608	SW,EFF	Tetrachloro-m-xylene	19-147
		Dibutylchlorendate	10-135
8081A	SED,S,HW	Tetrachloro-m-xylene	24-120
		Dibutylchlorendate	18-137
8082	SW, GW	Tetrachloro-m-xylene	19-147
8082	SED,S,HW	Tetrachloro-m-xylene	24-120

TABLE 5-39 cont

SURROGATE RECOVERY RANGES

METHOD	MATRIX	COMPOUND	RANGE
624	SW,EFF	1,2-Dichloroethane d4	70-131
		Toluene d8	83-115
		4-Bromofluorobenzene	73-119
		Ethylbenzene-d10	87-115
8260B	SW,GW	1,2-Dichloroethane d4	70-131
		Toluene d8	83-115
		4-Bromofluorobenzene	73-119
		Dibromofluoromethane	72-130
8260B	SED,S,HW	1,2-Dichloroethane d4	54-148
		Toluene d8	79-119
		4-Bromofluorobenzene	78-127
		Dibromofluoromethane	53-161
8021B	SW,GW	a,a,a-Trifluorotoluene	70-130
		Chloroprene	69-130
		1-Chloro-3-Fluorobenzene	65-132
		2-Chloropropane	61-132
8021B	S,SED,HW	a,a,a-Trifluorotoluene	60-140
		Chloroprene	63-139
		1-Chloro-3-Fluorobenzene	60-137
		2-Chloropropane	61-143

TABLE 5-39 cont

SURROGATE RECOVERY RANGES

METHOD	MATRIX	COMPOUND	RANGE
601	SW, EFF	Chloroprene	69-130
		1-Chloro-3-Fluorobenzene	65-132
		2-Chloropropane	61-132
602	SW,EFF	a,a,a-Trichlorotoluene	50-150
8015 High	SED,S,HW	Triacontane	51-123
8015 High	SW,GW	Triacontane	54-130
8015 Low	SED,S,HW	a,a,a,-Trifluorotoluene	50-150
8015 Low	SW, GW	a,a,a-Trifluorotoluene	50-150

6.0 SAMPLING PROCEDURES

The sample submitted to the laboratory must be collected in a manner such that it is representative of the larger source from which it was taken. The responsibility falls upon the person taking the sample to properly assess the site, choose the representative area to sample, collect the sample with appropriate implements, preserve, and transport the sample such that the representative nature of the sample remains intact. The laboratory sample for analysis will be a portion of the homogenous liquid or, if a solid, the aliquot will represent all layers of the sample.

Since our laboratory does not provide sample collection services, our responsibility in the sample collection process lies in suppling the sampler with the proper containers and preservatives. All containers used for the collection or transport of analytical samples are purchased "certified analytically clean".

6.1 SAMPLE CONTAINERS AND PRESERVATIVES

Table 6-1 describes the appropriate containers and preservatives required for the collection of various analytes. Bottles are purchased "certified clean" and the proper preservatives are added to the bottle prior to distribution to the sampling team. Trip Blanks for Volatiles are supplied.

All chemicals used as preservatives must be reagent grade or better. Nitric acid used to preserve samples for metals determination must be trace metal grade.

At the request of the client, preservatives may be supplied in separate containers for addition to the sample at the time of collection.

Certain states require special sampling protocols, e.g. Methanol preserved soils for Volatile Analysis. In these cases, the laboratory will supply the appropriate sampling containers, additional reagents such as methanol, and sampling instructions.

TABLE 6-1

SAMPLE REQUIREMENTS, STORAGE, AND HOLDING TIMES

PARAMETER	CONTAINER WATER	CONTAINER SOIL	ml WATER	gm SOIL	PRESERVATION WATER	PRESERVATION SOIL	HOLDING TIME WATER	HOLDING TIME SOIL
Acidity	P,G		50		Refrigerate		14 days	
Alklinity	P,G		50		Refrigerate		14 days	
Ammonia Nitrogen	P,G		100		Refrigerate pH <2 (S)		28 days	
BOD	P,G		1000		Refrigerate		48 hours	
COD	P,G		75		pH <2 (S)		28 days	
Chloride	P,G		50		Refrigerate		28 days	
Chlorine	P,G		100				Analyze Immediately	
Color	P,G		50		Refrigerate		48 hours	
Cyanide	P,G	4 oz	1000	100	Refrigerate ph >12 (N) 0.6g Ascorbic acid if free chlorine present	Refrigerate	14days	14 days
Fluoride	P		300		Refrigerate		28 days	
Kjeldahl Nitrogen	P,G		500		Refrigerate pH <2 (S)		28 days	
Metals	P,G	4 oz	200	100	pH < 2 (NI)	Refrigerate	180 days	6months
Mercury	P,G	4 oz	200	100	pH <2 (NI)	Refrigerate	28 days	28 days
Hexavalent Chrom	P,G		100		Refrigerate		24 hours	
Nitrate	P,G		100		Refrigerate		48 hours	
Nitrite	P,G		100		Refrigerate		48 hours	
Oil and Grease	G	4 oz	1000	100	Refrigerate pH <2 (S)	Refrigerate	28 days	14 days
Total Organic Carbon	P,G		50		Refrigerate pH <2 (S)		28 days	

TABLE 6-1 cont

SAMPLE REQUIREMENTS, STORAGE, AND HOLDING TIMES

PARAMETER	CONTAINER WATER	CONTAINER SOIL	ml WATER	gm SOIL	PRESERVATION WATER	PRESERVATION SOIL	HOLDING TIME WATER	HOLDING TIME SOIL
TRPH	G	4 oz	1000	100	Refrigerate	Refrigerate	28 days	28 days
Phenolics	G		500		Refrigerate pH <2 (S)		28 days	
Phosphorus	P,G		50		Refrigerate pH <2 (S)		28 days	
Ortho phosphorus	P,G		100		Refrigerate		48 hours	
Solids								
Total	P,G		100		Refrigerate		7 days	
Suspended	P,G		100		Refrigerate		48 hours	
Dissolved	P,G		250		Refrigerate		7 days	
Settleable	P,G		1000		Refrigerate		48 hours	
Volatile	P,G		100		Refrigerate		7 days	
Conductivity	P,G		100		Refrigerate		28 days	
Sulfate	P,G		100		Refrigerate		28 days	
Sulfide	P,G		500		Zinc Acetate + pH >9 (N)		7 days	
Turbidity	P,G		100		Refrigerate		48 hours	
Volatile Organics	VOA vial	4 oz	45	100	Refrigerate + HCl	Refrigerate	14 days	14 days
Acid Extractables	G	4 oz	1000	100	Refrigerate + 0.008% sodium Bisulfite	Refrigerate	7 d (ext) 40 d (anal)	14 d(ext) 40 d (a)
PCB's	G	4 oz	1000	100	Refrigerate	Refrigerate	7 d (ext) 40 d(A)	14 d(ext) 40 d(A)
BNA Extractable Org	G	4 oz	1000	100	Refrigerate	Refrigerate	7 d (ext) 40 d(A)	14 d(ext) 40 d(A)
Pesticides	G	4 oz	1000	100	Refrigerate	Refrigerate	7 d(ext) 40 d(A)	14 d(ext) 40 d(A)
Gross Alpha/Beta	P,G		1000		pH <2 (Nl)		180 days	

TABLE 6-1 cont

SAMPLE REQUIREMENTS, STORAGE, AND HOLDING TIMES

PARAMETER	CONTAINER WATER	CONTAINER SOIL	ml WATER	gm SOIL	PRESERVATION WATER	PRESERVATION SOIL	HOLDING TIME WATER	HOLDING TIME SOIL
Coliforms	P		100		Refrigerate Sodium bisulfite		8 hours	
pH	P,G	4 oz	100	100	Refrigerate	Refrigerate	24 hours	24 hours
Explosives	P,G	40 oz	1000	100	Refrigerate	Refrigerate	7 d(ext) 40 d(A)	14 d(ext) 40 d(A)

P = Plastic Container

G = Glass Container

N = Sodium hydroxide

NI = Nitric Acid

S = Sulfuric Acid

HCl = Hydrochloric Acid

Refrigerate = 1-4 degrees C

7.0 SAMPLE CUSTODY

It is our responsibility as a laboratory to insure the integrity of all samples received by our facility.

7.1 CHAIN OF CUSTODY DOCUMENTS

A document is required to certify that the sample has not been altered or contaminated. This document will detail the path that the sample travels from the time of collection until received by the laboratory. The names of persons who have custody of the sample will be carefully recorded as well as the inclusive times of possession. This will allow tracking of the sample from collection, through transport, to delivery to the laboratory.

A sample is said to be in the possession of an individual if:

1. It is in the actual physical possession of that person.

2. It is in view of that person after that person has had it in his physical possession.

3. It is secured in a restricted area.

All samples received should have a completed chain of custody with them when they are delivered to the laboratory. If the analysis is to demonstrate regulatory compliance or if the results are to be reported to any regulatory authority the samples must have a chain of custody associated with them. The chain of custody used by the laboratory is shown in figure 7-1 and should be completed as follows.

1. Each client should use pre-printed chain of custody forms.

2. The client will be responsible for the completion of the sample description and analysis requested components of the form. Date and time of collection should be completed.

3. The person or persons collecting the sample(s) must insure that all samples are uniquely identified and labeled with a sample description which is recorded on the chain of custody and also tagged onto the sample.

4. The person collecting the sample(s) begins the chain of custody by signing the block indicating that he was responsible for sample collection.

5. The sampler will sign the first "relinquished by" block when he transfers custody of the samples to the laboratory or courier.

6. The person transporting the samples will sign the "received" block and subsequently the "relinquished by" block as he transfers possession of the samples.

7. This process continues until the samples reach the laboratory log-in personnel where the person receiving the samples for the laboratory signs the "received by laboratory" block.

8. The log-in personnel will verify that all of the samples listed on the chain of custody were received and that the sample descriptions listed on the chain of custody match the sample descriptions on the sample containers. Any discrepancy between the containers and the chain of custody should be noted on the chain of custody. The log-in personnel will contact client services who will contact the client for resolution.

9. If samples are transported by commercial courier, e.g., Federal Express, the chain of custody and other papers should be sealed in a plastic bag and taped to the lid of the cooler. When the cooler is opened, the condition of the "custody seals" and the temperature of the samples is documented on the chain-of custody. Also a cooler receipt form will be completed if requested by the client.

10. The original chain of custody is kept on file in the laboratory. Copies are given to the client, given to client services for QC purposes, given to the Technical Support Manager for billing, and placed into the project folder for verification against the final report.

11. The samples are now the responsiblilty of the log-in supervisor who must maintain the security of the samples until analyzed. Archival of analyzed samples and final disposition is the responsibility of the Shipping Supervisor.

7.2 LOGGING OF SAMPLES INTO THE LABORATORY INFORMATION SYSTEM

The process of logging samples into the laboratory analytical system is the first critical step in an overall process that generates meaningful data. The process involves the following:

1. The temperature of the sample is taken and recorded on the chain of custody.

2. The integrity of each sample must be determined by comparing sample labels or tags with the chain of custody and by visual checks of the container for possible damage. Any problems that are noted are recorded on the chain of custody or on a separate sheet which is subsequently stapled to the chain of custody.

3. Samples requiring acid or base preservation shall be checked with pH paper to determine the effectiveness of the preservation. The pH of the sample is recorded on the chain of custody or, in the case of VOA's, the pH is recorded in a log book maintained by VOA lab personnel. Chlorine is checked on: Extractable Organics, BOD, TOX, Cyanide, Fluoride, Ammonia, TKN, Nitrate

4. Attention should be given to sampling times and dates. These dates are entered into the laboratory information system (LIMS). Specific due dates should also be entered. If there is a problem with the holding times of the samples the log- in supervisor will notify the client. If the holding times are short but not exceeded, the technical support manager should be notified so that processing can begin to meet holding times.

5. A sample may be composed of more than one bottle since different preservatives may be required to perform all analyses requested. A unique laboratory number is assigned by the LIMS. The LIMS will generate a sample label which is attached to each bottle of the sample. One of the labels is placed onto the chain of custody to complete the link between the original sampling document and the sample bottles. This log number will now serve to track and identify the sample throughout the laboratory.

6. Samples logged into the LIMS will have the following information entered:

   A. Laboratory Sample Number
   B. Sample Identification, i.e., sample description
   C. Client Name
   D. Date and Time Collected
   E. Date and Time Received by the Laboratroy
   F. Samplers Name
   G. Types and number of Sample Bottles Received
   H. Laboratory Project Number
   I. Analysis Requested
   J. The State in Which the sample was collected
   K. Sample Condition

7. If a sample is logged into the LIMS it is available for analysis. If a problem is noted that would prevent the analysis of that sample, e.g., broken bottles, the following procedure should be followed:

   A. Notify the client

   B. The client may cancel the request or agree to resubmit the request.

   C. All of the actions are recorded on the chain of custody or on a sheet which is attached to the chain of custody. The person making the final decision should be noted.

8. Samples are now transferred to the appropriate holding area until analysis. Volatiles are specifically stored in the isolated Volatile Laboratory with Refrigerator Blanks.

9. Sample creation logs are printed at the conclusion of the day and the data entered into the LIMS is checked against the chain of custody.

7.3 SAMPLE STORAGE

There are four primary concerns associated with sample storage: temperature, contamination, holding times, and security. Table 6-1 details the proper holding temperatures and holding times for various analyses perfomed by the laboratory.

Storage facilities can be secured; however, access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. The analytical departments receive an "incomplete" report each morning which lists all of the samples in the laboratory which have analyses in their departments still pending. By generating a worklist in the LIMS, all incompletes samples of a particular analyte are added to a lab batch for processing. This batch is given a worklist number which serves to track all QC related to the analytical batch.

Internal chain of custody forms may be required for specific projects. These will be generated by the sample log-in department once the samples are logged into the LIMS.

Once the analyst is finished with a sample it is returned to storage. When all requested analyses are completed the sample is transferred to sample archives. Samples are stored for three months before disposal. Sample storage is under the supervision of the Shipping Supervisor.

Extracts and digests of samples are to be treated as the sample itself. The digests/extracts should be maintained in secure areas within the appropriate analytical department.

Samples are stored in archives for 30 - 60 days before disposal (Contact Ohio VAP before disposal of Ohio Samples). The disposal of samples is in accordance with hazardous waste regulations and is monitored by the QA Officer. Extracts are kept for 30-60 days before disposal.

7.4 SAMPLE TRACKING

Samples are tracked within the laboratory using the main computer system. Work orders are entered into the lab system by log-in personnel. Worksheets are generated in the analytical department or the prep lab at the beginning of the analytical process. These worklists provide space for the time and date of analysis, analyst's signature, raw data, and calculations.

Once the worklist is completed the data is entered into the LIMS. The status of the sample may be monitored from any LIMS station by accessing the log number. Overdue and incomplete reports are monitored daily by the Technical Support Manager and the Laboratory Director.

An analytical batch may contain more than one client's samples and thus have more than one lab projects samples included in it. The raw data associated with the analytical batch is filed by worksheet name and date and time of completion.

Before final results are reported, Section 12 of this manual, the data is reviewed by the technical personnel of the department and the technical director or the QA Officer.

7.5 COMPUTER SECURITY

The LIMS uses a password security system. All personnel have a log-in name which restricts the access to system files based upon the level of security they are granted. All personnel are given a unique "Tech Number" which must be entered each time the system is accessed. This number tags each computer operation with the person initiating that operation, e.g., data entry or data modification. The following are the levels of security granted lab personnel:

A. Access to all files, including LIMS configuartion files and client data. Data modification and database modification is allowed.

B. Access to data entry and sample inquiry. No data modification allowed.

C. Review only. No entry of data or modification of data is allowed.

The Technical Director, QA Officer, Laboratory Director, and Laboratory Support Manager are assigned level A. Technical personnel are assigned level B. Clerical personnel are assigned security level C.

Reports are generated daily. Before the reports are generated the technical personnel have made all calculations and the data has been reviewed by a peer in the department. The worksheet is completed and data is entered into the LIMS. The LIMS generates an "echo" of the data entered worksheet which is verified against the manually created worksheet to prevent entry errors.

The completed worksheet, the "echo" worksheet, and the raw data are finally approved by the QA Officer, the Lab Director, or the Technical Director.

Final reports are reviewed against the chain of custody by the QA Officer, Laboratory Director, or Technical Director before the report is signed and released.

7.6 COMPUTER MAINTENANCE

The LIMS database is maintained by the Laboratory Director who is the system adminstrator. Technical support for the database system is provided by Northwest Analytical Systems. AIX and hardware support is provided by IBM. Daily, the LIMS data base is backed-up around noon and at the end of the working day. Once weekly, the entire LIMS system is backed-up and these tapes are stored in a secured vault off-site.

SPECIALIZED ASSAYS
ENVIRONMENTAL

Figure 7-1

7A- 009709

2960 Foster Creighton Drive
Nashville, TN 37204
615-726-0177
FAX 615/726-3404

REFERRING CLIENT

LLING CONTROL NUMBER (FOR LAB USE ONLY)

PROJECT #

P.O. #

IPLERS (Signature-Please Print)

PROJECT NAME

OR LAB USE ONLY ACC#	SAMPLE DESCRIPTION	DATE	TIME	COMP	GRAB	# OF CONT	ANALYSIS REQUESTED

elinquished by: (Signature)	Date / Time	Received by: (Signature)	Received for Laboratory by:	Date / Time
elinquished by: (Signature)	Date / Time	Received by: (Signature)	Remarks	
elinquished by: (Signature)	Date / Time	Received by: (Signature)		
ished by: (Signature)	Date / Time	Received by: (Signature)		

For further assistance in completing the chain of custody form please refer to the instructions found on the opposite s

8.0 ANALYTICAL PROCEDURES

8.1 APPROVED METHODS

Whenever possible the laboratory will use methods developed or approved by the USEPA. Sample type, source, and the governing regulatory agency requiring the analysis will determine the specific method chosen.

Laboratory Standard Operating Procedures (SOP's) have been developed for all analytical procedures. These procedures are formed around the appropriate EPA method and are adapted specifically to our laboratory. All SOP's are numbered sequentially and dated when approved for use in the lab. There will be no changes in the operating procedures without the written approval of the Technical Director and the Quality Assurance Officer. Any changes to the procedure must be made in blank ink and initalled by the Technical Director or the QA Officer. A change log will reflect the latest approved revision. The applicable portionof the SOP manual will be available in each department. Although the original EPA manual may be available, e.g., SW-846, no other SOP's are allowed. Elements of the methods contained in the SOP manual are:

1. Method name
2. References
3. Reagent and standard preparation
4. Analytical procedure (instrumentation, conditions, etc..)
5. Limitations of the procedure
6. Calibration
7. Calculations
8. Quality control

All methods must be apporved by the Technical Director and the Quality Assurance Officer before being placed into service.

8.2 PURCHASING OF REAGENTS

The nature of the analytical laboratory demands that all material used in any of the procedures be of a known quality. The wide variety of materials and reagents available makes it advisable to specify the name, brand, and grade of materials to be used in any determination. It will be the responsibility of the chemist to determine the quality of the materials needed if the quality is not specified in the SOP. The chemist should determine the vendor, make out a purchase request, and furnish that request to the purchasing department. The purchasing agent must have the approval of the Technical Director or the QA Officer before ordering any reagent chemical. It is the responsiblity of the purchansing agent to place the order, receive the shipment and date the material when received.

All material safety data sheets (MSDS) will be filed by the Safety Officer (QA Officer) of the laboratory. These sheets will be made available to all personnel for review.

All chemicals are dated upon receipt and should not be used past the expiration date if it listed. If no expiration date is listed reagent chemicals are considered suitable for use for one year after it is received.

8.3 REAGENT REQUIREMENTS

There are many different grades of analytical reagents available to the analytical chemist. All methods in use in the laboratory will specify the grade reagent that must be used in the procedure or process. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure, and, therefore, any grade reagent may be used. It is the responsibility of the analyst to carefully check the procedure and associated reagents to assure their suitability.

Reagents or working standards that are prepared in-house shall be dated, intialled by the analyst preparing the reagent and entered into the log book for tracking purposes. The tracking procedure for all standards requires that standards be given the identification A-B-C-D-E where A represents the standard log book number, B signifies that the standard is stock, working, intermediate, spiking, or surrogate. C represents the initials of the person preparing or receiving the standard material. D represents the page number of the log book where the entry was made, and E represents the entry number. Thus a standard may be designated 1-S-MD-59-100.

Only class "A" glassware shall be used in the preparation of any standard or reagent. Any material used in the preparation of a reagent must meet or exceed the quality of the standard or reagent chemical, e.g., solvents used in the preparation of organic standards.

All standard materials must be traceable to NBS/NIST standards, and records to that effect will be maintained in the area in which the standard is to be used.

Water used in the preparation of standards or reagents must be of laboratory grade type II. Water shall be considered type II if it has been processed through charcoal to remove organics and has passed through the laboratory reverse osmosis system and has a resulting conductance of less than 2.0 umhos. Records of the conductivity of the in-house prepared water shall be maintained. The Technical Support Manager must be immediately notified if the water exceeds specified limits. It will be the repsonsiblity of the Techical Support Manager to notify all analytical departments of an "out of specification" situation.

The laboratory may puchase reagent grade water for use in the determination of volatile organics. This water must be certified "organic free".

8.4 REAGENT STORAGE

The manner in which reagents and chemicals are stored is important from both the aspect of safety and reagent integrity. Generally, the following guidelines should be followed:

1. Light sensitive reagents should be stored in brown glass bottles.

2. Organic stock standards will be stored in a freezer.

3. Fresh solutions of working standards will be prepared from stock solutions and will be compared to the standard being replaced before placing it into service.

Table 8-1 gives specific storage instructions for reagents and chemicals used in the laboratory.

8.5 GLASSWARE

All volumetric glassware will be "Class A". Pyrex glass should be used where possible. For safety purposes, thick wall glassware should be used where available.

8.6 CLEANING OF GLASSWARE

The proper technique for cleaning glassware is chosen depending upon the intended use of the glassware being cleaned. The purpose of this is to remove all substances for the glassware that might interfere with the analysis. Water soluble substances can be removed with tap water followed with multiple rinses with laboratory grade water. In some instances

detergent may be required. Detergent washings should followed by two rinses with laboratory grade water.

Specific guidelines are:

Organic Extraction Glassware:

1. Rinse the glassware with isopropyl alcohol or acetone.

2. Wash the glassware with detergent (Chem-Solv or equivalent). A brush should be used to insure that all areas of the glassware have been thoroughly exposed to detergent.

3. Rinse with warm tap water followed by rinsing with laboratory grade water.

4. Dry the outside of the glassware with a towel.

5. Rinse the inside of the glassware with isopropyl alcohol or acetone. Rinse with extraction solvent immediately before use.

   Note: If visual inspection indicates remaining contamination, rinse the glassware in concentrated sulfuric acid and then repeat steps two through five. If the contamination remains discard the item.

Inorganic Glassware:

1. Wash the glassware in hot soapy water making sure all surfaces are covered.

2. Rinse well with warm tap water followed by two rinses with laboratory grade water.

3. For trace metal analysis, rinse/soak with nitric acid:water 1:1. Rinse with warm tap water.

4. Rinse/soak in 1:1 HCl/water, rinse with tap water followed by two rinses with laboratory grade water.

5. Glassware for phosphorus determination should be cleaned using the organic procedure.

Glassware Storage

1. Once cleaned, glassware should be capped or covered for storage in a cabinet.

2. Glassware must be stored away form any bulk chemicals or reagents.

8.7 WASTE DISPOSAL

The disposition of all wastes and samples is the responsiblity of the Quality Assurance Officer. The QA officer is responsbile for scheduling waste removal by the hazardous waste contractor and for maintaining records of types, volume, and date of disposal.

Organic Extraction Wastes

Waste organic solvent from organic extractions and glassware cleaning will be poured into drums labeled "WASTE FLAMMABLE LIQUID, N.O.S." It is the responsibility of the QA Officer to monitor the condition and capacity of these drums and schedule removal.

PCB Containing Wastes

Any organic solvent containing PCB's are to be segregated. Drums labeled "PCB CONTAINING SOLVENTS" are located in the waste storage area for the disposal of these wastes.

Sample Digests

Extracts and digests are held in archives for 30-60 days before final disposal. All extracts are combined and stored in approved waste containers prior to disposal by our contract waste disposal company.

Samples

On a case by case basis, samples may be returned to the client for disposal. Otherwise samples will be disposed of in accordance with current waste regulations. If a sample is known to be hazardous it must be placed into storage drums for disposal. Sample containers are crushed and the resulting glass waste is placed into drums for removal.

TABLE 8-1

STORAGE OF REAGENTS AND CHEMICALS

CHEMICAL	STORAGE REQUIREMENTS
Concentrated acids and bases	1
Standards for metals analysis	2
Standards for extractable organics	3
Standards for volatile organics	4
Bulk dry chemicals	5
Working solutions containing organic compounds	6
Working solutions containing only inorganics	7
Flammable solvents	8
Non-flammable solvents	9

STORAGE REQUIREMENT KEYS

1.0 Stored in the original containers in acid/base cabinets. All organics must be stored separately.

2.0 Stored at room temperature in the standards cabinet of the metals department.

3.0 Stored at temperatures below 0 degrees C in the department.

4.0 Neat standards are stored at room temperature in the standard cabinet in the department. Stock solutions and working solutions are stored in the freezer.

5.0 Bulk reagents are stored at room temperature in the reagent storage room of the laboratory.

6.0 Stored refrigerated at 1-4 degrees C in the departments.

7.0 Stored at room temperature in the department; refrigeration is optional.

8.0 Stored in solvent cabinets in the organic prep lab.

9.0 Stored separate from the flammable solvents in cabinets in the prep lab.

9.0 INSTRUMENT CALIBRATION

9.1 GENERAL OVERVIEW

Table 9-1 is a listing of the instrumentation used in the laboratory. All instruments used in the laboratory must be controlled with a formal calibration program. This program is integrated into the preventive maintenance program program of the department. This program will insure that instrumentation is operating within acceptable limits as required by the various methods.

Calibrations may be performed using reference standards, filters, etc., and may be performed by laboratory personnel or by the manufacturer's representative.

There are two types of calibration: operational and periodic. Operational calibration is performed as the instrument is used in an analysis and usually involves the development of response factors or curves relating to given amounts of standards. Periodic calibration should be performed on a prescribed time table and involves such actions as taking temperatures of ovens, baths, refrigerators, etc.

9.2 PERIODIC CALIBRATION

9.2.1 Procedures

Each instrument type will have a written protocol for periodic calibration. This procdedures will become a part of the laboratory SOP manual and will include the following:

1. Instrument description, including model number
2. The standards used for calibration if applicable
3. Performance tolerances
4. Performance frequency. This should be at least that recommended by the manufacturer.
5. Detailed procedure for calibration.
6. Instructions for proper action in response to unacceptable calibration responses.

Additionally, associated with each individual instrument, there will be records of calibration which will include the following:

1.0 Instrument model number

2.0 Standards used for calibration

3.0 Performance tolerances

4.0 Performance frequency

5.0 Results of the calibration, the initials of the individual making the calibration, and the date of the calibration

6.0 Records of calibration will be maintained for easy access and review. Table 9-2 is a summary of equipment that requires periodic calibration.

9.3 OPERATIONAL CALIBRATION AND STANDARDIZATION

For the purposes of this manual, calibration will refer to the qualitative aspects of instrument perfomance, e.g., GC/MS tuning, ICP profiling, or UV/VIS Spectrometer wavelength verification. Standardization will refer to the development of a quantitative relationship between instrumental response and concentration of particular analytes. The frequency of calibration and standardization is addressed in the methods being applied to the instrument.

9.3.1 Operational Calibration

Table 9-3 summarizes laboratory instrumentation that requires calibration for proper performance.

9.3.1.1 GC/MS Tuning

In order to obtain spectra that are comparable with those obtained from other instruments, the instrument should be tuned to deliver the same spectrum, both in mass numbers and intensities from a given compound. The compound used to establish the abundance criteria for base/neutral/acid organics (BNA's) is decafluorotriphenylphosphine, "DFTPP". For volatile organics, the tuning compound is bromofluorobenzene , "BFB". This calibration is required before standardization since standardization is directly

influenced by calibration.

1. DFTPP. Each GC/MS unit performing BNA analyses must be calibrated using DFTPP at the frequency mandated by the method, e.g., 625,8270C, etc. The abundance criteria of DFTPP is listed in table 9-4. The spectrum taken from the DFTPP analysis and used for calibration should be filed in both graphical and tabular formats.

2. BFB. Each GC/MS unit perfoming VOA analyses must be calibrated using BFB at the frequency mandated by the method, e.g., 624, 8260B. The abundance criteria of BFB is listed in table 9-5. A bar graph and a tabular listing of the ion abundances should be filed with all related data sheets.

9.3.1.2 Inductively Coupled Plasma Emissions Spectrometer Profiling

The ICP must be calibrated against a mercury lamp each day and interelement correction factors verified using an interference check solution, Table 9-10.

9.3.2 Operational Standardization

Table 9-11 lists instrument standardization requirements.

9.3.2.1 Standards

All standards used in the laboratory must be tracked from the time of purchase or preparation through the analysis. Standards purchased from outside vendors must be traceable to NBS/NIST, and certifying documentation must be filed in the department purchasing the standard. All stock standards are assigned a unique identification number and entered into the log book when prepared or placed into use. All intermediate and working standards prepared from the stock standards will be logged into the standard log book with the concentration of the diluted standard, the date, the name of the analyst preparing the standard, and log number of the stock standard from which it was prepared. Standard sources and storage requirements are listed in Table 9-6.

9.3.2.2 GC/MS Standardization

Following calibration of the instrument using BFB or DFTPP, the instrument must be standardized against reference solutions of five concentrations. The concentration of these standards is specific to the method. Additionally, for VOA's, three internal standards and three surrogate compounds are added immediately prior to analysis. Table 9-7 lists the internal standards and some of the compounds to which they relate.

The BNA instruments must be standardized against injections of 10, 20, 50, 80, and 100 ng of the compounds being determined. Six internal standards are used in the BNA analysis. Table 9-8 lists the BNA internal standards and some of the associated analytes.

The method being followed specifies the acceptance criteria for the standardization. Response factors are generated for each compound:

$$Rf = \frac{Ax}{Ais} \times \frac{Cis}{Cx}$$

Rf = response factor
Ax = area of the characteristic ion for the compound
Ais = area of the characteristic ion for the internal standard
Cis = concentration of the internal standard (ng/ul)
Cx = concentration of the compound being determined

SW-846 methods require that certain "calibration check compounds" (CCC's) listed in Table 9-9 must fall within certain prescribed limits for the standardization to be considered acceptable. Method 624 requires that all target compounds have a %RSD of <30.

The relative standard deviation (RSD) must be calculated for all compounds:

$$\%RSD = \left(\frac{\delta}{X}\right) 100$$

RSD = relative standard deviation

δ = standard deviation of initial five response factors

X = mean of initial five response factors

For SW-846 methods the %RSD of the CCC's must be less than 30% in order to accept the calibration

In addition to checking the statistics of the curve, the instrument repsonse must be checked by checking the "system performance check" compounds (SPCC's). The required minimum response for these compounds is listed in Table 9-9.

Once the five point calibration has been completed, a check of the validity of the standardization must be made using a "verification" standard. This standard is analyzed and quantified against the five point calibration curve. The values obtained must be within 20% of the true value for the standardization to be acceptable. No analysis can begin until all standardization processes are completed and successful.

9.3.2.3 Standardization of the Gas Chromatograph

Before beginning an analysis using the gas chromatograph a five point standardization curve must be run with the initial standard having a concentration near the detection limit of the analysis being performed. The response factors are calculated for each compound at each concentration level and these factors are averaged. The mean response factor is used to calculate concentrations of the compounds being determined. The %RSD must be less than 20% for these respnse factors. The continuiing standardization standard must fall within 15% of the average response factor for the compounds being analyzed. If unknown samples exceed the range of the standard curve they must be diluted and reanalyzed.

9.3.2.4 Standardization of the ICP

The ICP is standardized by introducing into the plasma solutions containing the metals being determined. A calibration blank and one standard (10 mg/L) is sufficient for standardization. An independent standardization verification solution and an initial calibration blank are analyzed immediately following standardization. "Continuing calibration verification" standards (CCV's) and "continuing calibration blanks" are anaylzed after every ten samples and at the end of the analytical batch. The CCB's must never exceed the reporting limit for that element.

The CCV's must always fall within 10% of the true value else all samples back to the last acceptable CCV and CCB must be repeated since the system is considered to be out of calibration.

To assess the effects of inter-element interferences on selected elements an "interference

check sample" (ICS) must be analyzed at the beginning and end of each run. Table 9-10 lists the interferents and the affected elements along with the concentrations of each element in the ICS.

The determined value of each of the affected elements must be within 20% of the true value else the run is terminated and an evaluation of the inter-element correction factors is made. The ICS is also checked for negative values; acceptance criteria is +/- 10% referenced to blank values.

The linearity of the ICP must be determined quarterly. This is accomplished by increasing the concentration of solutions being aspirated into the instrument until a 10% difference between the true value and the determined value is noted. That concentration is the upper limit of linearity for that channel.

9.3.2.5 Standardization of the Atomic Absorption Spectrometer

The atomic absorption spectrometer must be standardized with a blank and three standards for each analytical run. The curve fit for the calibration must exceed 0.995. The validity of the standards used for calibration must be shown be running an independent "initial calibration verification" standard (ICV) before any analyses can begin. This standard must fall within 10% of its true value to be within acceptable limits. The "initial calibration blank" (ICB) is run following standardization to demonstrate that the instrument is free of contamination. This blank must not exceed the reporting limit for the element. "Continuing calibration blanks" (CCB's) must be analyzed every ten samples. The CCV must fall within 10% of the expected value of the calibration or must be considered invalid.

TABLE 9-1

LABORATORY INSTRUMENTATION

ORGANICS

1	Varian 3700 GC with FID and ECD detectors
1	Varian 3700 GC with ECD and ECD detectors
1	Varian 3400 GC with FID detector
1	Perkin Elmer Autosys GC with ECD and ECD detectors
4	Perkin Elmer Autosys GC with FID detector
7	Tremetrics (540 or 9001) GC with PID and FID detectors and Purge / Trap
1	Tremetrics 9001 GC with ECD and ECD and NP detectors
1	Tremetrics 9001 GC with PID and ELCD detectors and Purge / Trap
1	Tracor 540 GC withPID and ELCD detectors and Purge / Trap
1	Tracor 540 GC withPID and FID detectors and Purge / Trap
2	Hewlett-Packard 5890 GC with PID and FID detectors and Purge / Trap
4	Hewlett-Packard, GC/MS MSD-5900 series with Purge / Trap
3	Hewlett-Packard, GC/MS MSD-5900 series with autosampler
2	Shimadzu SCL-6B dual pump HPLC with UV and Fluorescence detectors
4	Refrigerators

METALS

1	ICP Jarrell Ash 61E 32 simultaneous channels
1	ICP Jarrell Ash Trace 32 simultaneous channels
1	Atomic Absorption Perkin Elmer 1100
1	Atomic Absorption Perkin Elmer 5100
1	Atomic Absorption Perkin Elmer 4100
2	Mercury Analyzer, Leeman PS-200
2	Microwave Digestion Units, CEM
3	Fume Hoods
2	Refrigerators

TABLE 9-1 cont

GENERAL CHEMISTRY, INORGANICS

1	Flow Injection, Lachat 8000
1	Ion Chromatograph, Dionex QIC
1	Calorimeter, Parr
1	pH Meter, Corning
1	pH Meter, Orion
1	Total Organic Carbon (TOC), Shimadzu 5050
1	Total Organic Halogen (TOX), Dohrman
1	Total Organic Halogen (TOX), Mitsubishi
1	Turbidimeter, HF-DRT-100
1	Specific Conductance, Orion 126
1	Fluorometer, Sequoia-Turner 450
1	UV/Visible Spectrometer, Shimadzu
1	Midi-Distillation, Andrews Glass
1	Midi-Distillation, Westco
1	Block Digestor, Lachat 60011
1	Flash Point, Pensky Martens
1	Flash Point, Koehler
1	COD Reactor, Hach
1	Karl Fisher Titrator, Brinkmann
1	Oxygen Probe, Orion
1	Furnace, Thermolyne 1400
1	Alpha / Beta Counter, Tennelec 5100
2	BOD Incubators, Fisher 307
1	Solvent Evaporator, Thermolyne
2	Ovens MB-2729Q
1	Oven Grieve 323
1	Furnace, Fisher 186A
4	Fume Hoods
2	Refrigerators

TABLE 9-1 cont.

PREP. LABORATORY, ORGANIC / TCLP

1	Infrared (IR), Perkin Elmer 1600, FTIR
2	Ovens, Blue M
1	Balance, Mettler AE240
1	Balance, Denver Instruments XS410
2	Centrifuge, Fisher 228
1	Centrifuge, DYNAC
1	Centrifuge, Clay Adams
1	Water Chiller, NESLAB
2	Sonicator, Heat systems
4	Sonicator, Tekmar
2	Ultra Sonic Bath, Fisher-28
12	ZHE Rotators, Millipore
1	ZHE Pressure, Millipore
4	Shakers, Eberbach
2	pH Meter, Accumet
1	pH Meter, Corning
2	Rotators for TCLP, Associated Design
20	Hot Plates, Thermolyne
13	Fume Hoods

LOG-IN / SAMPLE RECEIPT

1	Fume Hood
1	Cold Storage Room
1	2 Refrigerators for Volatiles Only

TABLE 9-1 cont.

AIR / INDUSTRIAL HYGIENE LABORATORY

1 Varian 3400 Gas Chromatograph with FID
1 Varian 3400 Gas Chromatograph equipped with FID and Tedlar Bag Autosampler
1 Entech 7000 Summa Cannister Pre-Concentrator with Hewlett-Packard 5971 Gas chromatograph / Mass Spectrometer

TABLE 9-2

PERIODIC CALIBRATION

INSTRUMENT	TYPE CALIBRATION	FREQUENCY
Analytical balances	Accuracy determined using Class S weights	daily
Refrigerators	Temperature checked using Calibrated thermometers	twice daily
Spectophotometers	Wavelength accurcy and absorbance Accuracy check using certified standard solutions	six months
Conductivity meters	Cell impedance determination with KCl solution	each use
Ovens	Temperature checked using calibrated thermometers	daily
Mass Spectrometers	Mass Calibration	as needed

TABLE 9-3

OPERATIONAL CALIBRATIONS

INSTRUMENT	TYPE CALIBRATION	REQUIREMENTS	FREQUENCY
TOX Analyzer	Verification of silver titration cell	Titrated standard must fall with 2% of true value	Each batch
Mass Spectrometer	Mass assignment and Abundance tuning	DFTPP for semivolatiles BFB for volatiles	Every 12 hours
ICP	Wavelength accuracy	Mercury 253.7 line must be centered within the 0.2 nm slit	Daily

TABLE 9-4

DFTPP KEY IONS AND ABUNDANCE CRITERIA

MASS	ION ABUNDANCE
51	30-60 % of mass 198
68	Less than 2% of mass 69
70	Less than 2% of mass 69
127	40-60% of mass 198
197	Less than 1% of mass 198
198	Base ion, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	Greater than 1% of mass 198
441	Present but less than mass 443
442	Greater than 40% of mass 198
443	17-23% of mass 442

TABLE 9-5

BFB KEY IONS AND ABUNDANCE CRITERIA

MASS	ION ABUNDANCE
50	15-40% of mass 95
75	30-60% of base peak
95	Base peak, 100% relative abundance
96	5-9% of base peak
173	Less than 2% of mass 174
174	Greater than 50% of base peak
175	5-9% of mass 174
176	95-101% of mass 174
177	5-9% of mass 176

TABLE 9-6

STANDARD SOURCES AND PREPARATON

INSTRUMENT	SOURCE	HOW RECEIVED	STORAGE	STOCK	STORAGE	FREQ
ICP	SPEX	1000 ppm solutions	Room temp	Working stds prepared from Stock	Room temp	daily
AA	Perkin-Elmer	1000 ppm solutions	Room temp	Intermediate stds from stock	Room temp	bi-monthly
				Working stds prepared from intermediate stds	Room temp	daily
GC	Supelco Ultra	Solutions	Refigerated	Intermediate stds from stock	Room temp	monthly
				Working stds prepared from intermediate stds	Room temp	monthly
TOX	Dohrman	Solutions	Refrigerated	Working stds from stock	Refrigerate	monthly
TOC	Fisher/ Dohrman	Solutions	Refrigerated	As received	Refrigerate	N/A
GC/MS	Ultra	Solutions	Refrigerated	Working std from stock	Refrigerate	monthly
Infrared	Aldrich Sigma	Pure reagent	Room Temp	Working std from stock	Refrigerate	weekly

TABLE 9-7

INTERNAL STANDARDS FOR VOLATILE ORGANICS

PENTAFLUOROBENZENE

Dichlorodifluoromethane
Chloromethane
Vinyl Chloride
Bromomethane
Chloroethane
Trichlorofluoromethane
Acetone
1,1-Dichloroethene
Methylene Chloride
Carbon Disulfide
t-1,2-Dichloroethene
Methyl-t-Butyl Ether
1,1-Dichloroethane
Vinyl Acetate
2-Butanone
c-1,2-Dichloroethene
Bromochloromethane
Chloroform
2,2-Dichloropropane
1,2-Dichloroethane
1,1,1-Trichloroethane
1,1-Dichloropropene

1,4-DIFLUOROBENZENE

Carbon Tetrachloride
Benzene
Dibromomethane
1,2-Dichloropropane
Trichloroethene
Bromodichloromethane
2-Chloroethylvinylether
c-1,3-Dichloropropene
4-Methyl-2-Pentanone
t-1,3-Dichloropropene
1,1,2-Trichloroethane
Toluene
1,3-Dichloropropane
2-Hexanone

CHLOROBENZENE-d5

Dibromochloromethane
1,2-Dibromoethane
Tetrachloroethene
1,1,1,2-Tetrachloroethane
Chlorobenzene
Ethylbenzene
m,p-Xylene
Bromoform
Styrene
o-Xylene
1,1,2,2-Tetrachloroethane
1,2,3-Trichloropropane

1,4-DICHLOROBENZENE-d4 & ETHYLBENZENE-d10

Isopropylbenzene
Bromobenzene
Propylbenzene
2-Chlorotoluene
4-Chlorotoluene
1,3,5-Trimethylbenzene
t-Butylbenzene
1,2,4-Trimethylbenzene
sec-Butylbenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
p-isopropyltoluene
Butylbenzene
1,2-Dibromo-3-Chloropropane
1,2,4-Trichlorobenzene
Naphthalene
Hexachlorobutadiene
1,2,3-Trichlorobenzene

TABLE 9-8

INTERNAL STANDARDS FOR SEMIVOLATILE ORGANICS

1,4-DICHLOROBENZENE	NAPHTHALENE D8	ACENAPTHENE D10
N-Nitrosodimethylamine	Nitrobenzene	Hexachlorocyclopentadiene
Phenol	Isophorone	2,4,6-Trichlorophenol
Aniline	2-Nitrophenol	2,4,5-Trichlorophenol
bis-2-Chloroethylether	2,4-Dimethylphenol	2-Chloronaphthalene
2-Chlorophenol	Benzoic Acid	2-Nitroaniline
1,3-Dichlorobenzene	bis-2-Chloroethoxymethane	Dimethylphthalate
1,4-Dichlorobenzene	2,4-Dichlorophenol	Acenapthylene
Benzyl Alcohol	1,2,4-Trichlorobenzene	3-Nitroaniline
1,2-Dichlorobenzene	Naphthalene	Acenaphthene
2-Methylphenol	4-Chloroaniline	2,4-Dinitrophenol
bis-2-Chloroisopropylether	Hexachlorobutadiene	4-Nitrophenol
3-Methylphenol	2-Methylnaphthalene	Dibenzofuran
N-Nitroso-di-N-propylamine	Nitrobenzene d5	2,4-Dinitrotoluene
Hexachloroethane		2,6-Dinitrotoluene
2-Fluorophenol		Diethylphthalate
Phenol d5		4-Chlorophenylphenylether
		Fluorene
		4-Nitroaniline
		2-Fluorobiphenyl
		2,4,6-Tribromophenol

PHENANTHRENE D10	CHRYSENE D12	PERYLENE D12
4,6-Dinitro-2-methylphenol	Benzidien	Di-n-octylphthalate
N-Nitrosodiphenylamine	Pyrene	Benzo(b)fluoranthene
1,2-Diphenylhydrazine	Butylbenzylphthalate	Benzo(k)fluoranthene
4-Bromophenylphenylether	3,3'-Dichlorobenzidine	Benzo(a)pyrene
Hexachorobenzene	Benzo(a)anthracene	Indeno(1,2,3-cd)pyrene
Pentachlorophenol	bis-2-Ethylhexylphthalate	Dibenz(ah)anthracene
Anthracene	Chrysene	Benzo(ghi)perylene
Di-n-butylphthalate	Terphenyl d14	
Fluoranthene		

TABLE 9-9

CALIBRATION CHECK COMPOUNDS

BASE/NEUTRAL FRACTION	ACID FRACTION	VOLATILES
Acenapthene	4-Chloro-2-methylphenol	1,2-Dichloroethene
1,4-Dichlorobenzene	2,4-Dichlorophenol	Chloroform
Hexachlorobutadiene	2-Nitrophenol	1,2-Dichlorpropene
N-Nitroso-di-n-propylamine	Phenol	Toluene
di-n-Octylphthalate	Pentachlorophenol	Ethylbenzene
Benzo(a)pyrene	2,4,6-Trichlorophenol	Vinylchloride

SYSTEM PERFORMANCE CHECK COMPOUNDS

BASE NEUTRAL FRACTION	MINIMUM RF
N-Nitroso-di-n-propylamine	0.05
Hexachlorocyclopentadiene	0.05
ACID FRACTION	
2,4-Dinitrophenol	0.05
4-Nitrophenol	0.05
VOLATILES	
Chloromethane	0.30
1,1-Dichloroethane	0.30
Bromoform	0.25
1,1,2,2-Tetrachloroethane	0.30
Chlorobenzene	0.30

TABLE 9-10

INTERFERENCE CHECK SAMPLE ELEMENT CONCENTRATIONS

SOLUTION A		SOLUTION B	
ANALYTES	MG/L	INTERFERENT	MG/L
Ag	1.0	Al	500
Ba	0.5	Ca	500
Be	0.5	Fe	200
Cd	1.0	Mg	500
Co	0.5		
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	1.0		
V	0.5		
Zn	1.0		
As	1.0		
Se	1.0		
Tl	1.0		
Sb	1.0		

TABLE 9-11

INSTRUMENT STANDARDIZATION

INSTRUMENT	# Stds INIT CAL	Acceptance CRITERIA	FREQUENCY	# Stds CONT CAL	Acceptance CRITERIA	FREQ
GC-pesticides 8081A	5	%RSD <20	Failure of CCV	1	Must be within 15%	10%
608;8082	3	%RSD <10	Failure of CCV	1	Must be within 15%	Daily
Herbicides 8151A	5	%RSD <20	Failure of CCV	1	Must be within 15%	10%
Total Org Hal	1	Within 5% of true	Each Batch	1	Must be within 5% of true	20%
Total Org Carbon	4	R = >0.99	Failure of CCV	1	Must be within 5% of true	10%
COD	5	R = >0.99	Failure of CCV	1	Must be within 20% of true	Batch
BOD	1	200 +/- 40	Each Batch	N/A	N/A	N/A
Ion Chrom	4	R = >0.99	Failure of CCV	1	Must be within 5% of true	10%
GC/MS VOA's 8260B	5	RSD of CCC's must be < 30%. SPCC's must have minimum Rf of 0.3 with exception of Bromoform at 0.25	Failure of daily or continuing	1	CCC's RSD must be <25% SPCC's must meet min calibration	12 hr Rf.
624	3	RSD of all targets <35	Failure of daily calibration check	1	QC Check standard must be within limits	Daily
GC/MS BNA's 8270C	5	CCC's RPD must be <30%. SPCC's must have Rf > 0.050	Failure of daily or continuing cal	1	CCC's must be <25% SPCC's RF must be > 0.050	12 hr
625	3	RSD of all targets <35	Failure of daily Calibration check	1	QC check standard must be within limits	Daily
ICP 6010B	1	Independent cal std	Daily	1	Must be within 10% of true	10%
ICP 200.7	1	Independent cal std	Daily	1	Must be within 5% of true	10%
AA	3	R = >0.99	Each run	1	Must be within 10% of true	10%
Infrared	5	R= >0.995	Failure of CCV	1	Must be within 15%	10%

10.0 PREVENTIVE MAINTENANCE

Preventive maintenance is the action taken to maintain proper instrument function and perfomance, e.g., cleaning, adjustment, lubrication, etc. To be considered within the preventive maintenance program are:

1. Instrument manufacturers' suggested maintenance schedule.

2. Appropriate spare parts inventory.

3. Frequency and assignment preventive maintenance.

The section supervisor is responsible for the preventive maintenance of instruments within his department. Documentation of all maintenance, scheduled or unscheduled, will be kept for each instrument. Log books will be assigned to each instrument in which will be recorded maintenance, date performed, and the name of the individual performing the maintenance. These records become part of the laboratory's permanent record.

NOTE: Some daily/routine maintenance will not be recorded, e.g., GC Septa change.

Table 10-1 lists laboratory instrumentation and the required maintenance for each instrument. Items requiring recording in the logbook are noted.

10.1 CONTINGENCY PLANS

Even with redundant instrumentation, there are instrumental failures that require immediate action to prevent loss of irreplaceable samples due to holding time expiration. The following steps should be followed subsequent to any instrument failure:

1. Immediately notify the section supervisor. It is the supervisor's responsibility to assess the severity of the failure and to estimate the expected down time.

2. If, in the judgement of the supervisor, the instrument will not be operational in time to perform the analyses scheduled on that instument within the allowed time, the analyses will be shifted to other instrumentation if available.

3. If the repair is judged to be outside of the abilities of the laboratory personnel, the Operations Manager is notified and a call is placed for manufacurer's service.

4. The Operations Manager is responsible for evaluating sample load and holding times. If, in his judgement, projects will be delayed, clients are immediately notified. If, in his judgement, the holding times for the samples will be expire before analysis will be completed, the client is notified and arrangements are made to refer the samples to other laboratories for analysis.

5. The Technical Support Manager must be kept apprised of the instrument failure in order to prevent scheduling of further analyses on the instrument.

TABLE 10-1

PREVENTIVE MAINTENANCE

INSTRUMENT	ACTIVITY	FREQUENCY
Gas Chromatograph	Change Septum	Daily
	Check gases	Daily - record
	Repack or Replace Column	As needed - record
	Clean Detector	As needed - record
	Check autosampler seals	Daily
	Clean injectors; replace liners	As needed - record
	Clean PID lamp	Daily
HPLC/IC	Check Seals for Leakage	Each use
	Replace seals/valves/lamps	As needed - record
	Replace suppressor	As needed - record
GC/MS	Change Septum on GC	Daily
	Bake Trap	Daily
	Clean Source	Tune Failure - record
	Change pump oil	Quarterly - record
	Clean injector/replace liner	SPCC failure - record
ICP	Torch Inspection	Each use
	Clean Torch and Nebulizer	As needed - record
	Inspect Filters	Daily
	Change Filters	As needed - record
	Inspect Pump Tubing	Daily
	Change Pump Tubing	As needed - record
Atomic Absorption	Inspect Graphite Tube	Daily
	Inspect Contact Rings	Daily
	Clean Windows	Daily
	Align Lamp	Each Use
UV/VIS	Check Paper	Daily
	Clean Sample Compartment	As needed
	Auto-check Calibration	Daily at start up
	Wavelength cal	Every six months

TABLE 10-1 cont

INSTRUMENT	ACTIVITY	FREQUENCY
Infrared	Change desicant	As needed - record
Mercury Analyzer	Change Drying Tubes	Daily
	Run Aperture test	Daily
	Inspect Tubes and reagents	Daily
Conductivity Meter	Clean cell	Each use
	Calibrate Cell	Each use
Turbidimeter	Check lamp	Each use
	Clean Sample Holder	Each use
pH Meter	Clean Electrode	Each use
	Inspect Electrode	Each use
Total Organic Carbon Analyzer	Check Gas flow	Daily
	Check fluid level	Daily
	Replace "O" rings	As needed - record
	Check needle	Daily
	Replace Scrubbers	Yearly-record
	Replace catalyst	As needed - record
Total Organic Halogen	Clean Inlet tube	As needed
	Clean cell	As needed
Fluorometer	Clean cells	Each use
Calorimeter	Calibrate thermometer	Yearly

TABLE 10-1 cont

INSTRUMENT	ACTIVITY	FREQUENCY
Temperature Devices Refrigerators	Temperature	Daily or when used (Refrigerators 2X/day)
Incubators, BOD Evaporators Flash Tester COD Reactor Water Circulator Drying Ovens		
Weighing Balances	Clean pan Check calibration	Each use Daily - record
Ultra-sonic disruptors	Clean	Each use
Zero Headspace Extractors	Verify Rotation Speed Check for leakage	Yearly Each use
TCLP Extractors	Verify Rotation speed	Yearly

11.0 ASSESSMENT OF ACCURACY AND PRECISION

11.1 QUALITY CONTROL CHECK

The performance of all analytical methods must be monitored to assess the accuracy and reproducibility of the procedure. Specific quality control checks are designed to provide the necessary information for method assessment.

The basic quality control unit is the sample batch. A sample batch is defined as a group of samples of a similar matrix, analyzed in the same method sequence, using the same lots of reagents, and processed at the same time. A batch size is controlled by the method; however, it is never greater than 20 samples.

Quality control samples are defined below. Methods requiring various quality control samples, their purpose, and concentrations are listed in Table 11-1.

11.1.1 Blanks

Blanks are artificial samples of an analytical matrix designed to detect the introduction of artifacts into the system.

11.1.1.1 Method Blank

The method blank is prepared from an aliquot of analyte free matrix that is processed through the entire analytical process and is analyzed with the sample set. One method blank is analyzed per sample batch or one in twenty samples whichever is more frequent.

11.1.1.2 Analytical (reagent) Blank

The analytical blank is prepared from laboratory grade water processed through the analytical system and analyzed with the sample set. The analytical blank may substitute for the method blank if an interference free matrix is not available.

11.2.2 Spiked Samples

Samples fortified to a known and validated concentration of analytes being determined are termed matrix spikes. The relationship between the known concentration and the

analyzed value is termed the "percent recovery." Where sufficient sample is available, a matrix spike (and duplicate where required by the procedure) is analyzed in each sample batch or one sample in twenty, whichever is more frequent.

11.1.2.1 Reagent Spikes

Reagent spikes are prepared by fortifying an analyte-free matrix with analytes prior to preparation.

11.1.2.2 Matrix Spikes

Matrix spikes are prepared by fortifying a sample chosen from the sample batch with all analytes being determined.

11.1.2.3 Surrogate Spikes

Compounds having similar chemical characteristics to those being analyzed but which are not generally found in environmental samples are used as surrogates compounds. Known concentrations of these compounds are added to all samples in the batch prior to sample preparation.

11.1.3 Quality Control Checks

Samples or standards from an independent source used to verify calibration, standardization, and procedural accuracy and precision are quality control samples.

11.1.3.1 Calibration verification standards

Standard solutions from a source other than the source of the original standardization materials are used to verify standardization.

11.1.3.2 Quality control check samples

Samples obtained from an independent source for which the concentrations of the analytes have been validated are termed quality control check samples. These samples are introduced into the analytical stream by the QA Officer and are analyzed as "blind" samples. These samples are analyzed semi-annually.

11.1.3.3 Laboratory control samples

Samples obtained from an independent source for which the concentrations of the analytes have been established are used as laboratory control samples. These samples are introduced into the analytical stream and are analyzed as required by the method.

11.2.3 Duplicates

In order to assess procedural precision, two aliquots of the same same sample are prepared and analyzed in the same batch. Some procedures require the sample to be split and spiked in duplicate, as in 11.1.2.2. Duplicates are assayed with each sample batch or one in twenty samples as required by the method.

11.2 ROUTINE METHODS TO ASSESS PRECISION AND ACCURACY

Accuracy and precision of laboratory methods are assesed by statistically analyzing the data from matrix spike and duplicate analyses. Table 11-1 specifies the procedures and concentrations used to develop accuracy and precision data.

11.2.1 Accuracy

The percent recovery of each matrix spike is calculated as:

$$\frac{ssr - sr}{sa} \quad X \quad 100 \quad = \quad \text{Percent Recovery}$$

Where: ssr = analyte concentration in spiked sample

sr = analyte concentration is original sample

sa = spike added

When at least thirty samples have been analyzed, the mean percent recovery and the standard deviations are calculated:

$$\%R_m = \frac{\Sigma \%R ..... \%R_n}{n}$$

$$S=\sqrt{\frac{\%R_n-\%R_m}{n-1}}$$

Where: %Rm = mean percent recovery
% Rn = the percent recovery of a single pair
n = the number of results
S = standard deviation of the data set percentage recovery

The accuracy of the method is expressed as the target mean and a range of three standard deviations from the mean. Quality control data is entered into the LIMS where it is tabulated and a running mean and target range is calculated. At least 30 data points are used to calculate statistics.

Quality control charts are accessible through the computer. The acceptable range of a calculated recovery data point may be determined by comparing the value to the mean and three standard deviation range listed on the chart. These charts may be displayed or printed at any time. The QA Officer will print and review the charts for trends or other QC anomalies.

11.2.2 Precision is accesed by the statistical analysis of data from the duplicate analyses

$$\%RPD = \frac{D_1-D_2}{(D_1+D_2)/2}X100$$

When at least thirty samples have been analyzed the mean and standard deviation is calculated:

$$M = \frac{m_1 \ldots . m_n}{n}$$

$$S_m = \sqrt{\frac{(m-M)^2}{n-1}}$$

Where:
m = the RPD of a replicate pair
M = the average of the RPD determinations
Sm = the standard deviation of the data set RPD determinations
n = the number of determinations

The acceptable range for the precision is the mean +/- 3 standard deviations. Quality control charts are generated by the computer and reviewed by the QA director monthly. The mean and standard deviation is calculated after each data point is entered.

11.3 METHOD DETECTION LIMITS AND PRACTICAL QUANTITATION LIMITS

11.3.1 Method detection limits

The method detection limit is the smallest concentration of analyte that can be measured and reported within 99 percent confidence that the concentration is greater than zero. MDL's are determined by the procedure outlined in Appendix B 40 CFR, part 136.

Laboratory grade water is fortified with analytes at a concentration 1-10 times the

estimated method detection limit. Seven consecutive aliquots of the sample are taken through the entire analytical sequence. The variance and standard deviation of the replicate measurements are determined as follows:

$$S^2 = \frac{1}{n-1}[\Sigma_{i=1}^{n} x_i^2 - (\Sigma_{i=1}^{n} x_i)^2/n]$$

Where:

$\Sigma_{i=1}^{n} x_i$ = Sum of all values in the final reporting units from the initial through the final analysis.

The MDL is computed by multiplying the standard deviation by the student's "t" value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom and the "t" value at 99% confidence level would be 3.143 when n = seven.

The MDL's are verified and updated annually.

11.3.2 The practical quantitation limit is the smallest concentration of analyte that can be reported with some degree of confidence. PQL's are generated from the same data used to determine the MDL's. The PQL may vary on a project by project basis, but it is generally 5-10 times the MDL.

TABLE 11-1

QUALITY ASSURANCE TARGETS

PARAMETER	PURPOSE	CONCENTRATION LEVEL	METHOD REFERENCES
Duplicates	Precision	Low	All 200 and 7000 series AA Methods. 200.7 and 6010 For ICP
Duplicate Matrix Spikes	Precision and Accuracy	Low	600 Series organics 8000 Series organics 300 and 900 Series anions 330.5 and 9010 cyanide 9020 TOX 376.1 and 9030 sulfide 415.1 and 9060 TOC 420.1 and 9065 phenolics
Matrix Spikes	Accuracy	Low Mid	All Furnace AA metals All ICP metals
Lab Control Samples	Accuracy	Mid	All AA and ICP metals All volatile and extractable Organics

Low concentration level: Lower 20% of the linear range
Mid concentration level: 20 - 80% of the linear range
High concentration level: 80 - 100% of the linear range

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

12.1 DATA REDUCTION

It is the responsibility of the analyst to perform the procedures strictly according to the written protocols found in the Standard Operating Procdure manual of the laboratory. This includes the analysis of proper QC materials, standards, and blanks. The analyst is further responsible for the accuracy of the test result and the recording of all related data on the appropriate worksheet. Some instruments have the option of direct "upload" of instrument data into the LIMS.

The results of QC materials must be compared to acceptable ranges and any outliers must be flagged. Once all calculations have been made, QC results evaluated, and all data recorded, the raw data is again reviewed by the supervisor of the section.

The LIMS generated worksheet is used to enter the data into the LIMS. Once the worksheet is completed, the LIMS will print an "echo" worksheet containing the information input on all of the samples of the batch. This sheet should be compared to original worksheet to insure that no data entry errors were made. The person perfoming the analysis, the person inputting the data into the LIMS, and the person reviewing the entry must all initial the first sheet of the worksheet. These steps also apply to direct "upload" of instrument data into the LIMS.

The final review of the data is made by the Laboratory Director, the QA Officer, or the Technical Director. Both worksheets, the original and the echo, and the raw data are included in the review package. The person completing the final review must initial the data set.

The data set to be reviewed will include all of the following:

1. Worksheets with all related calculations.
2. All instrument charts, chromatograms, and printouts; if data was uploaded into the LIMS, then a print out of the electronic data is also included.

12.2 DATA CALCULATIONS

Final results are calculated differently depending upon the method chosen to generate the data. Methods with associated calculation types are listed in Table 12-1. Final results may be calculated by the LIMS given the extract/digest concentration, the dilution factors, and the original weight/volume of sample extracted or digested. The analyst always validates the final sample calculations.

12.3 DATA VALIDATION

The process of data validation involves various checks from the evaluation of sample integrity to verification of quality control values. Tables 12-2 and 12-3 lists the steps of data validation and the personnel responsible for each action. Table 12-4 lists the quality control procedures and acceptable ranges.

12.4 DATA REPORTING

All analysts in the laboratory are assigned numbers which are used to access LIMS. When entering data this number is tagged to the data and becomes a part of the permanent record for that sample. Once all data on a project is entered the LIMS will print the final report. All data is now flagged as complete and is tagged with the laboratory project number. The folder containing all data relevant to the project and the final report are now reviewed by the Laboratory Manager, the QA Officer, or the Technical Director. Once the final project review is complete and the project is found to be satifactory, the reviewer signs the final report.

12.5 CORRECTION OF ERRONEOUS DATA

If it is determined that erroneous data has been released to the client, the following procedure must be followed to correct the report:

1. The Laboratory Director or the QA Officer is notified

2. The Laboratory Director, the QA Officer or their designee will notify the client by phone

3. There will be an explanation of the change included in the project folder.

4. The data in the LIMS is changed by the QA Officer, the Laboratory Director, or their designee.

5. A written explanation of the error will be submitted to the QA Officer and a copy to the Technical Director. Once these have been reviewed and the Technical Director's copy returned to the QA Officer, a copy is filed with the project data.

6. A corrected report is generated and the client notified.

12.6 DATA STORAGE

All data relating to an analytical project is stored for seven years (Records on Ohio Samples-10 years). Computer records are archived onto magnetic tape. Electronic records of instrument outputs are stored in the department and are filed by date.

TABLE 12-1

CALCULATIONS

PROCEDURE	CALCULATION TYPE	CALIBRATION	MODE
GC/MS SW-846	5-Point calibration	linear regression	Computer
600 series	3-Point calibration	Linear regression	Computer
AA metals	3-Point calibration	linear regression	Computer
ICP metals	Single point		Computer
GC Organics	5-Point calibration	linear regression	Computer
TRPH-IR	5-Point calibration	linear regression	Computer
Cyanide	5-Point calibration	linear regression	Computer
Phenolics	3-Point calibration	linear regression	Computer
Hardness	Direct titration		Manual
Alkalinity	Direct titration		Manual
BOD	Direct readout		Manual
COD	3-Point calibration	linear regression	Computer
Phosphate	5-Point calibration	linear regression	Computer
Ammonia N	5-Point calibration	linear regression	Computer
TOC	4-Point calibration	linear regression	Computer
Solids	Direct determination		Manual
MBAS	10-Point calibration	linear regression	Computer
HPLC	5-Point calibration	linear regression	Computer

TABLE 12-2

DATA INTEGRITY

ACTION	PERSONS RESPONSIBLE
Verification of sample integrity	Log-in personnel
Chain of Custody verification	Log-in personnel
Check of sample appropriateness	Analyst/Log-in personnel
Checking of extraction logs	Analyst
Verification of instrument calibration	Analyst
Checking of raw data and calculations	Section supervisor and peer review
Checking of instrument logs	Section supervisor
Internal chain of custody (if applicable)	Section supervisor
Completeness	QA Officer, Laboratory Director, or Technical Director

TABLE 12-3

DATA VALIDATION

ACTION	PERSON RESPONSIBLE
Review of project completeness	Technical Director Quality Assurance Officer Laboratory Director
Review of quality control	Quality Assurance Officer Operations Manager
Review of supporting documentation	Operations Manager
Final project review	Technical Director Quality Assurance Officer Laboratory Director

TABLE 12-4

QUALITY CONTROL PROCEDURES

QUALITY CONTROL PROCEDURES	ACCEPTABLE RANGE
Duplicates	RPD must be within three standard deviations of the mean
Matrix Spikes	Recovery must be within the limits stated in section 13.
Method Blanks	Results for the method blank cannot exceed the project required reporting limit
Surrogates	Surrogate recoveries must be within three standard deviations of the mean
Laboratory Control Samples	The analytically determined result must be within twenty percent of the true value unless otherwise stated in section 13.

If the acceptance criteria for the duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. If the LCS is within acceptable limits the batch is acceptable. The results of the duplicates, matrix spikes, and the LCS are reported with the data set.

If the method blank is out of acceptable limits the entire batch must be repeated.

If surrogates are out of limits the individual sample must be repeated.

13.0 CORRECTIVE ACTION

The acceptance criteria for quality control procedures, along with associated corrective actions, is indicated in Tables 13-1 through 13-11. During method perfomance the analyst may perform corrective action as required as it relates to calibration, standardization, or repeating the analysis. Supervisor approval is required for corrective action involving the re-preparation of extracts, preparation of new standards, purchase of new standards, determining or accepting data involving interferences, and making requests for manufacturer's service.

Corrective action may be initiated by external sources. Unacceptable performance evaluations by regulatory agencies, poor correlation on field split samples, external audits (performance or system), or questions concerning data quality by clients will lead to investigation and corrective action.

All corrective action results will be documented. If the supervisor of the analytical area performed the corrective action, e.g., initiation of the purchase of new standard materials, then the supervisor will document the department records of his action. If the corrective action is initiated externally, the action will be followed to compeletion and documented by the QA Officer. The following information will be documented as part of the correction action:

1. Regulatory Agency
2. Item number or lab sample number
3. Results reported and the associated disrepancy
4. Criteria for acceptibility
5. Action taken
6. Signatures of all involved individuals

Corrective action will always be initiated when recommended by quality assurance agencies such as the Corps of Engineers, the USEPA, or state regulatory agencies.

TABLE 13-1

QUALITY CONTROL CRITERIA AND CORRECTIVE ACTION

METHOD NUMBER	ANALYSIS	PARAMETER	CRITERIA	CORRECTIVE ACTION FOR FAILURE
608	Pest, PCB's	QC Check Standards	If MS is outside ranges in Table 13-4 QC Check Std must be analyzed and fall within those ranges	Repeat sample set
		Initial Calibration	Minimum of 3 standards, one of which must be near the MDL. RSD <10% Use averarge RF or best fit curve if r > 0.99.	Repeat calibration
		Continuing Calibration	One or more standards analyzed daily. Must be within 15% of expected value.	Repeat initial cal
		Matrix Spikes	One MS per 10 samples from one site or one per month which ever is more frequent. Must be within ranges in Table 13-4.	Run QC Check Std
8081A/8082		QC Check Standard	Is MS/MSD fall outside ranges listed in Table 13-5 analyze QC Check Std. Values must meet those ranges.	Repeat sample set
		Initial Calibration	Minimum of 5 standards, the lowest of which must be near the MDL. RSD must be < 20%. Use average RF or best fit curve if r > 0.99.	Repeat calibration
		Continuing Calibration	Mid-level standard must be run every 10 samples. Must be within 15% of true.	Repeat initial cal
		Surrogates	Two, within 3 SD of mean	Repeat sample

TABLE 13-1 cont

METHOD NUMBER	ANALYSIS	PARAMETER	CRITERIA	CORRECTIVE ACTION FOR FAILURE
8081A/8082 continued		MS/MSD	Analyze on MS and MSD per 20 samples or per prep batch which ever is more frequent. Ranges must fall within those of Table 13-5.	Run QC Check Std
		Method Blanks	One blank per batch or 20 samples. Must be < CRDL.	Repeat sample batch
		Breakdown	Total breakdown of DDT and Endrin < 20%	Repeat sample batch
625	Semi-VOA's	QC Check Standard	If MS falls outside of ranges listed in Table 13-6 QC Check Standard is analyzed and must fall within those ranges.	Repeat sample set
		Initial Calibration	Three standards, one near the MDL. %RSD < 35. Use average RF.	Repeat calibration
		Continuing Calibration	One or more standards are analyzed each day. Must be within 20% of expected.	Repeat initial cal
		Surrogate Standards	Minimum of three. Must fall within +/-2SD.	Repeat Sample
		Matrix Spike	One per 20 samples from each site or one per month which ever is more frequent. Compare to ranges in Table 13-6.	Run QC Check Std
		Method Blank	One blank per batch. Must be < CRDL	Repeat sample set

TABLE 13-1 cont

METHOD NUMBER	ANALYSIS	PARAMETER	CRITERIA	CORRECTIVE ACTION FOR FAILURE
625 cont		DFTPP	Must meet criteria in Table 13-3	Must be met before Beginning analysis
		DDT Breakdown	Must be < 20%	Must be met before Beginning analysis
8270C	Semi-VOA's	QC Check Standard	If MS/MSD fall outside ranges of Table 13-7, QC Check Standard must be analyzed and fall within those ranges.	Repeat calibration
		Initial Calibration	Minimum of 5 standards, the lowest of which must must be near the MDL. %RSD for CCC's must be < 30. RF for SPCC's must be >0.05.	Repeat calibration
		Continuing Calibration	Mid-level standard run every 12 hours. RF for SPCC's > 0.05. RF of CCC's must be <30% difference from initial calibration.	Repeat initial cal
		Surrogate Standards	Recovery must be within +/- 3SD of lab determined values.	Repeat sample
		MS/MSD	One set per batch or every 20 samples. Values must be within those of Table 13-7.	Run QC Check Std
		Method Blanks	One blank per batch or every 20 samples. Must be < CRDL.	Repeat sample set
		DFTPP	Must meet criteria in Table 13-3	Must be met before Beginning analysis

TABLE 13-1 cont

METHOD NUMBER	ANALYSIS	PARAMETER	CRITERIA	CORRECTIVE ACTION FOR FAILURE
8270C cont		DDT Breakdown	Must be < 20%	Must be met before Beginning analysis
624	VOA's	QC Check Standard	If MS results fall outside values listed in Table 13-8 QC Check Standard must be analyzed and fall within those ranges.	Repeat sample set
		Initial Calibration	Minimum of three standards, the lowest of which must be near the MDL. %RSD must be <35. Use average RF.	Repeat calibration
		Continuing Calibration	QC Check Standard daily. Compare to Table 13-8.	Repeat Initial Cal
624		Surrogate Standards	Minimum of three. Must be within +/- 3SD of lab established limits.	Repeat sample
		Matrix Spikes	One MS per 20 samples. Values must fall within those of Table 13-8	Run QC Check Std
		Method Blanks	One blank per day. Must be < CRDL.	Repeat sample set
		BFB Tuning	Must meet criteria in Table 13-2	Must be met before beginning analysis
8260B	VOA's	QC Check Standards	If MS/MSD falls outside ranges of Table 13-9, QC Check Standard must be analyzed and fall within those ranges.	Repeat sample batch
		Initial Calibration	Minimum of 5 standards. Lowest must be near MDL. %RSD for CCC's < 30.	

TABLE 13-1 cont

METHOD NUMBER	ANALYSIS	PARAMETER	CRITERIA	CORRECTIVE ACTION FOR FAILURE
8260B cont			RF for SPCC's > 0.30 (Bromoform 0.25)	Repeat calibration
		Continuing Calibration	Mid-level standard every 12 hours. %D for CCC's must be <20%; RF for SPCC's >0.30 (0.25 for bromoform).	Repeat Calibration
		Surrogate Standards	Three. Recovery limits must be within +/- 3SD	Repeat sample
		MS/MSD	One set per batch or every 20 samples. Must meet criteria of Table 13-9.	Run QC Check Std
		Method Blanks	One per batch. Must be < CRDL.	Repeat sample set
		BFB Tuning	Must meet criteria of Table 13-2	Must be met before beginning analysis
601,602	GC VOA's	QC Check Standard	If MS is outside limits of Table 13-10, QC Check Standard must be run and fall within those limits.	Repeat sample set
		Initial Calibration	Minimum of three stds, the lowest of which must be near the MDL. %RSD must be < 10.	Repeat calibration
		Continuing Calibration	QC Check Standard analyzed daily. Compare to values in Table 13-10.	Repeat initial cal
		Surrogate Standards	Must be within laboratory established ranges.	Repeat sample
		Matrix spike	One MS per 10 samples. Acceptable ranges in Table 13-10.	Run QC Check Std

TABLE 13-1 cont

METHOD NUMBER	ANALYSIS	PARAMETER	CRITERIA	CORRECTIVE ACTION FOR FAILURE
601/602 cont		Method Blanks	One per day. Must be < CRDL.	Repeat sample set
8021B	GC VOA's	QC Check Standard	If MS/MSD are outside of ranges listed in Table 13-11, QC Check Std must be run and fall within those ranges.	Repeat sample set
		Initial Calibration	Mimimum of 5, the lowest of which must be near the MDL. %RSD must be less than 20.	Repeat calibration
		Surrogate Standards	Recoveries must fall within +/- 3SD.	Repeat sample
		Continuing Cal	Mid-level standard run every 20 samples. Must be within 15% of expected value.	Repeat initial cal
		MS/MSD	One MS/MSD per batch. Acceptable ranges are listed in Table 13-11.	Run QC Check Std
		Method Blanks	One per batch. Must be < CRDL.	Repeat sample set
335.2	Cyanide	Initial Calibration	Six standards. r > 0.99	Repeat calibration
		Matrix Spike	> 90% recovery	Repeat sample set
9010	Cyanide	QC Check Standard	Must be within 15% of true value.	Repeat calibration
		Initial Calibration	Six standards. r > 0.99	Repeat calibration
		Continuing Calibration	Verify curve with a mid-level standard. Must be within 15% of true.	Repeat calibration

TABLE 13-1 cont

METHOD NUMBER	ANALYSIS	PARAMETER	CRITERIA	CORRECTIVE ACTION FOR FAILURE
415.1, 9060	TOC	Initial Calibration	Four standards. r > 0.99	Repeat calibration
		QC Check Standard	Verify standard curve Must be +/- 15% of true	Repeat initial cal
		Continuing Cal	Run mid-level standard every 20 samples. Must be +/- 15 % of true.	Repeat sample set; initial calibration
245.5	Mercury	Initial Calibration	Six standards plus blank, r > 0.99	Repeat calibration
		Continuing Calibration	Run a low-level std every 20 samples. Must be +/- 10%.	Recalibrate
7471	Mercury	QC Check Standard	Verify standard curve with an independent standard. Must be +/- 10%.	Recalibrate
		Initial Calibration	Five standards plus a blank. r > 0.99	Recalibrate
		Continuing Cal	Run a mid-level std every 10 samples. Must be +/- 20%.	Recalibrate
		Method Blank	One per batch. Must be less than CRDL.	Repeat sample batch
200	AA Metals	Initial Standardization	Three standards plus blank. r > 0.99	Repeat calibration
		Continuing Standards	Analyze one low-level standard every 20 samples. Must be +/- 10%.	Repeat calibration

TABLE 13-1 cont

METHOD NUMBER	ANALYSIS	PARAMETER	CRITERIA	CORRECTIVE ACTION FOR FAILURE
7000	AA Metals	QC Check Standard	Verify calibration with an independent check std. Must be within 10%.	Repeat calibration
		Initial Calibration	Three standards plus blank. r > 0.99	Repeat calibration
		Continuing Calibration	Mid-level standard every 10 samples. Must be within 20%.	Repeat calibration
		MS/MSD	One per batch. Recovery 75-125%.	Analyze LCS
		LCS	If MS/MSD fails, LCS must be within %15 of true value.	Repeat batch
200.7	ICP Metals	QC Check Standards	Verify calibration with independent standard. Must be within 5%.	Recalibrate
		Continuing Calibration	Mid-range standard at beginning, every 10 samples and at end of run. Must be within 5%.	Recalibrate
		Method Blank	One per batch. Must be < CRDL.	Recalibrate
		Continuing Cal Blank	Analyze calibration blank every 10 samples. Must be less than CRDL.	Recalibrate
6010B	ICP Metals	Initial Cal Verification	Verify standard curve. Must be within 10 %.	Recalibrate

TABLE 13-1 cont

METHOD NUMBER	ANALYSIS	PARAMETER	CRITERIA	CORRECTIVE ACTION FOR FAILURE
6010B cont		Continuing Calibration	After every 10 samples and at the end of the run. Must be within 10%.	Recalibrate
		MS/MSD	Once set per batch. Must be +/- 2SD	Analyze LCS
		LCS	Analyze if MS/MSD fails. Must be within 10%.	Repeat batch
		Method Blank	One per batch. Must be less than CRDL.	Repeat batch
		Continuing Cal Blank	Analyze every 10 samples and at the end of the run. Must be < CRDL.	Repeat batch
		Interference Check	Analyze at the beginning and end of each run. Must be within 20%.	Check interelement correction factors.

TABLE 13-2

BFB KEY IONS AND ABUNDANCE CRITERIA

MASS	ION ABUNDANCE
50	15-40 % of the base peak
75	30-60- % of the base peak
95	Base peak, 100%
96	5-9 % of the base peak
173	Less than 2 % of mass 174
174	Greater than 50 % of the base peak
175	5-9 % of mass 174
176	95-101 % of 174
177	5-9 % of 176

TABLE 13-3

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

MASS	ION ABUNDANCE CRITERIA
51	30-60 % of mass 198
68	Less than 2 % of 69
70	Less than 2 % of 69
127	40-60 % of 198
197	Less than 1% of 198
198	Base ion
199	5-9 % of 198
275	10-30% of 198
365	Greater than 1 % of 198
441	Present but less than mass 443
442	Greater than 40 % of 198
443	17-23 percent of 442

TABLE 13-4

QC ACCEPTANCE CRITERIA FOR METHOD 608

PARAMETER	CONC ug/l	RANGE ug/l
Adrin	2.0	1.08-2.24
a-BHC	2.0	0.98-2.44
b-BHC	2.0	0.78-2.60
g-BHC	2.0	1.01-2.37
d-BHC	2.0	0.86-2.32
Chlordane	50	27.6-54.3
4,4'-DDD	10	4.8-12.6
4,4'-DDE	2.0	1.08-2.60
4,4'-DDT	10	4.6-13.7
Dieldrin	2.0	1.5-2.49
Endosulfan I	2.0	1.14-2.82
Endosulfan II	10	2.2-17.1
Endsosulfan sulfate	10	3.8-13.2
Endrin	10	5.1-12.6
Heptachlor	2.0	0.86-2.00
Heptachlor epoxide	2.0	1.13-2.63
Toxaphene	50	27.8-55.6
PCB 1016	50	30.5-51.5
PCB 1221	50	22.1-75.2
PCB 1242	50	24.8-69.6
PCB 1248	50	29.0-70.2
PCB 1254	50	22.2-57.9
PCB 1260	50	18.7-54.9

TABLE 13-5

QC ACCEPTANCE CRITERIA FOR METHOD 8081A/8082

PARAMETER	CONC ug/l	RANGE
Adrin	2.0	1.08-2.24
a-BHC	2.0	0.98-2.44
b-BHC	2.0	0.78-2.60
g-BHC	2.0	1.01-2.37
d-BHC	2.0	0.86-2.32
Chlordane	50	27.6-54.3
4,4'-DDD	10	4.8-12.6
4,4'-DDE	2.0	1.08-2.60
4,4'-DDT	10	4.6-13.7
Dieldrin	2.0	1.5-2.49
Endosulfan I	2.0	1.14-2.82
Endosulfan II	10	2.2-17.1
Endsosulfan sulfate	10	3.8-13.2
Endrin	10	5.1-12.6
Heptachlor	2.0	0.86-2.00
Heptachlor epoxide	2.0	1.13-2.63
Toxaphene	50	27.8-55.6
PCB 1016	50	30.5-51.5
PCB 1221	50	22.1-75.2
PCB 1242	50	24.8-69.6
PCB 1248	50	29.0-70.2
PCB 1254	50	22.2-57.9
PCB 1260	50	18.7-54.9

TABLE 13-6

QC ACCEPTANCE CRITERIA FOR METHOD 625

PARAMETER	CONC ug/l	RANGE
Acenaphthene	100	60.1-132.3
Acenaphthylene	100	53.5-126
Aldrin	100	7.2-152.2
Anthracene	100	43.4-118
Benzo(a)anthracene	100	41.8-133
Benzo(b)fluoranthene	100	42.0-140.4
Benzo(k)fluoranthene	100	25.2-145.7
Benzo(a)pyrene	100	31.7-148.0
Benzo(ghi)perylene	100	D-195.0
Butylbenzyphthalate	100	D-139.9
b-BHC	100	41.5-130.6
g-BHC	100	D-100.0
Bis(2-chloroethyl)ether	100	42.9-126
Bis(2-chloroethoxy)methane	100	49.2-164.7
Bis(2-chloroisopropyl)ether	100	62.8-138.6
Bis(2-ethylhexyl)phthalate	100	28.9-136.9
4-Bromophenylphenyl ether	100	64.9-114.4
2-Chloronaphthalene	100	64.5-113.5
4-Chlorophenyl phenyl ether	100	38.4-144.7
Chrysene	100	44.1-139.1
4,4'-DDD	100	D-134.5
4,4'-DDE	100	19.2-119.7
4,4'-DDT	100	D-170.6
Dibenzo(a,h)anthracene	100	D-199.7
Di-n-butylphthalate	100	8.4-111.0
1,2-Dichlorobenzene	100	48.6-112.0
1,3-Dichlorobenzene	100	16.7-153.9
1,4-Dichlorobenzene	100	37.3-105.7
3,3'-Dichlorobenzidine	100	8.2-212.5
Dieldrin	100	44.3-119.3
Diethyl phthalate	100	D-100.0
Dimethyl phthalate	100	D-100.0
2,4-Dinitrotoluene	100	47.5-126.9

TABLE 13-6 cont

QC ACCEPTANCE CRITERIA FOR METHOD 625

PARAMETER	CONC ug/l	RANGE
2,6-Dinitrotoluene	100	68.1-136.7
Di-n-octyl phthalate	100	18.6-131.8
Endosulfan Sulfate	100	D-103.5
Endrin aldehyde	100	42.9-121.3
Fluorene	100	71.6-108.6
Heptachlor	100	D-172.2
Heptachlor epoxide	100	70.9-109.4
Hexachlorobenzene	100	7.8-141.5
Hexachlorobutadiene	100	37.8-102.2
Hexachloroethane	100	55.2-100.0
Indeno(1,2,3-cd)pyrene	100	D-150.9
Isophorone	100	46.8-180.2
Naphthalene	100	35.6-119.6
Nitrobenzene	100	54.3-157.6
N-nitroso-di-n-propylamine	100	13.6-197.9
PCB 1260	100	19.3-121
Phenanthrene	100	65.2-108.7
Pyrene	100	69.6-100.0
1,2,4-Trichlorobenzene	100	57.3-129.2
4-Chloro-3-methylphenol	100	40.8-127.9
2-Chlorophenol	100	36.2-120.4
2,4-Dichlorophenol	100	52.5-121.7
2,4-Dimethylphenol	100	41.8-109.0
2,4-Dinitrophenol	100	D-172.9
2-Methyl-4,6-dinitrophenol	100	53.0-100.0
2-Nitrophenol	100	45.0-166.7
4-Nitrophenol	100	13.0-106.5
Pentachlorophenol	100	38.1-151.8
Phenol	100	16.6-100.0
2,4,6-Trichlorophenol	100	52.4-129.2

TABLE 13-7

QC ACCEPTANCE CRITERIA FOR METHOD 8270C

PARAMETER	CONC ug/l	RANGE
Acenaphthene	100	60.1-132.3
Acenaphthylene	100	53.5-126
Aldrin	100	7.2-152.2
Anthracene	100	43.4-118
Benzo(a)anthracene	100	41.8-133
Benzo(b)fluoranthene	100	42.0-140.4
Benzo(k)fluoranthene	100	25.2-145.7
Benzo(a)pyrene	100	31.7-148.0
Benzo(ghi)perylene	100	D-195.0
Butylbenzyphthalate	100	D-139.9
b-BHC	100	41.5-130.6
g-BHC	100	D-100.0
Bis(2-chloroethyl)ether	100	42.9-126
Bis(2-chloroethoxy)methane	100	49.2-164.7
Bis(2-chloroisopropyl)ether	100	62.8-138.6
Bis(2-ethylhexyl)phthalate	100	28.9-136.9
4-Bromophenylphenyl ether	100	64.9-114.4
2-Chloronaphthalene	100	64.5-113.5
4-Chlorophenyl phenyl ether	100	38.4-144.7
Chrysene	100	44.1-139.1
4,4'-DDD	100	D-134.5
4,4'-DDE	100	19.2-119.7
4,4'-DDT	100	D-170.6
Dibenzo(a,h)anthracene	100	D-199.7
Di-n-butylphthalate	100	8.4-111.0
1,2-Dichlorobenzene	100	48.6-112.0
1,3-Dichlorobenzene	100	16.7-153.9
1,4-Dichlorobenzene	100	37.3-105.7
3,3'-Dichlorobenzidine	100	8.2-212.5
Dieldrin	100	44.3-119.3
Diethyl phthalate	100	D-100.0
Dimethyl phthalate	100	D-100.0
2,4-Dinitrotoluene	100	47.5-126.9

TABLE 13-7 cont

QC ACCEPTANCE CRITERIA FOR METHOD 8270C

PARAMETER	CONC ug/l	RANGE
2,6-Dinitrotoluene	100	68.1-136.7
Di-n-octyl phthalate	100	18.6-131.8
Endosulfan Sulfate	100	D-103.5
Endrin aldehyde	100	42.9-121.3
Fluorene	100	71.6-108.6
Heptachlor	100	D-172.2
Heptachlor epoxide	100	70.9-109.4
Hexachlorobenzene	100	7.8-141.5
Hexachlorobutadiene	100	37.8-102.2
Hexachloroethane	100	55.2-100.0
Indeno(1,2,3-cd)pyrene	100	D-150.9
Isophorone	100	46.8-180.2
Naphthalene	100	35.6-119.6
Nitrobenzene	100	54.3-157.6
N-nitroso-di-n-propylamine	100	13.6-197.9
PCB 1260	100	19.3-121
Phenanthrene	100	65.2-108.7
Pyrene	100	69.6-100.0
1,2,4-Trichlorobenzene	100	57.3-129.2
4-Chloro-3-methylphenol	100	40.8-127.9
2-Chlorophenol	100	36.2-120.4
2,4-Dichlorophenol	100	52.5-121.7
2,4-Dimethylphenol	100	41.8-109.0
2,4-Dinitrophenol	100	D-172.9
2-Methyl-4,6-dinitrophenol	100	53.0-100.0
2-Nitrophenol	100	45.0-166.7
4-Nitrophenol	100	13.0-106.5
Pentachlorophenol	100	38.1-151.8
Phenol	100	16.6-100.0
2,4,6-Trichlorophenol	100	52.4-129.2

TABLE 13-8

CALIBRATION AND QC ACCEPTANCE CRITERIA FOR METHOD 624

PARAMETER	RANGE ug/l
Benzene	12.8-27.2
Bromodichloromethane	13.1-26.9
Bromoform	14.2-25.8
Carbon tetrachloride	14.6-25.4
Chlorobenzene	13.2-26.8
Chloroethane	7.6-32.4
2-Chloroethylvinyl ether	D-44.8
Chloroform	13.5-26.5
Chloromethane	D-40.8
Dibromochloromethane	13.5-26.5
1,2-Dichlorobenzene	12.6-27.4
1,3-Dichlorobenzene	14.6-25.4
1,4-Dichlorobenzene	12.6-27.4
1,1-Dichloroethane	14.5-25.5
1,2-Dichloroethane	13.6-26.4
1,1-Dichloroethene	10.1-29.9
T-1,2-Dichloroethene	13.9-26.1
1,2-Dichloropropane	6.8-33.2
C-1,3-Dichloropropene	4.8-35.2
T-1,3-Dichloropropene	10.0-30.0
Ethylbenzene	11.8-28.2
Methylene chloride	12.1-27.9
1,1,2,2-Tetrachloroethane	12.1-27.9
Tetrachloroethene	12.1-27.9
Toluene	14.9-25.1
1,1,1-Trichloroethane	15.0-25.0
1,1,2-Trichloroethane	14.2-25.8
Trichloroethene	13.3-26.7
Trichlorofluoromethane	9.6-30.4
Vinyl chloride	0.8-39.2

*** Based upon a 20 ug/l target

TABLE 13-9

CALIBRATION AND QC ACCEPTANCE CRITERIA FOR METHOD 8260B

PARAMETER	RANGE ug/l
Benzene	77.5-116.5
Bromodichloromethane	77.9-112.1
Bromoform	84.8-117.2
Carbon tetrachloride	61.8-106.2
Chlorobenzene	73.6-108.4
Chloroethane	65.0-113.0
2-Chloroethylvinyl ether	D-44.8
Chloroform	73.5-106.5
Chloromethane	68.1-117.9
Dibromochloromethane	82.2-115.8
1,2-Dichlorobenzene	86.5-107.5
1,3-Dichlorobenzene	83.0-119.0
1,4-Dichlorobenzene	86.5-125.5
1,1-Dichloroethane	80.7-111.3
1,2-Dichloroethane	79.7-110.3
1,1-Dichloroethene	75.1-112.9
t-1,2-Dichloroethene	77.4-108.6
1,2-Dichloropropane	79.3-114.7
c-1,3-Dichloropropene	76.1-124.2
t-1,3-Dichloropropene	76.1-124.2
Ethylbenzene	83.4-114.6
Methylene chloride	80.0-110.0
1,1,2,2-Tetrachloroethane	64.0-136.0
Tetrachloroethene	81.0-111.0
Toluene	82.3-117.7
1,1,1-Trichloroethane	74.3-121.7
1,1,2-Trichloroethane	87.3-116.7
Trichloroethene	70.5-109.5
Trichlorofluoromethane	67.4-110.6
Vinyl chloride	78.5-117.5

Based upon a 100 ug/l target

TABLE 13-9 cont.
Method 8260B

PARAMETER	RANGE ug/l
Dichlorodifluoromethane	72.6-125.4
Bromomethane	71.6-118.4
Methyl-t-Butyl Ether	N/A
c-1,2-Dichloroethene	80.9-121.1
Bromochloromethane	72.9-107.1
2,2-Dichloropropane	42.2-129.8
1,1-Dichloropropene	71.9-124.1
Dibromomethane	83.2-116.8
1,3-Dichloropropane	81.6-116.4
1,2-Dibromoethane	80.2-113.8
1,1,1,2-Tetrachloroethane	85.9-114.1
m,p-Xylene	78.7-115.3
o-xylene	83.5-128.5
Styrene	39.0-153.0
1,2,3-Trichloropropane	76.5-115.5
Isopropylbenzene	82.8-121.2
Bromobenzene	74.8-119.2
Propylbenzene	79.2-118.8
2-Chlorotoluene	85.2-112.8
4-Chlorotoluene	75.0-117.0
1,3,5-Trimethylbenzene	88.4-113.6
t-Butylbenzene	102.-117.5
sec-Butylbenzene	88.7-131.3
p-Isopropylbenzene	74.0-152.0
Butylbenzene	76.0-112.0
1,2-Dibrom-3-Chlorpropane	62.0-122.0
1,2,4-Trichlorobenzene	48.0-139.0
Naphthalene	76.4-119.6
Hexachlorobutadiene	79.9-120.1
1,2,3-Trichlorobenzene	75.3-128.7

Based upon a 100 ug/l target

TABLE 13-10

QC ACCEPTANCE CRITERIA FOR METHODS 601/602

PARAMETER	RANGE ug/l
Bromodichloromethane	15.2-24.8
Bromoform	14.7-25.3
Bromomethane	11.7-28.3
Carbon tetrachloride	13.7-26.3
Chlorobenzene	14.4-25.6
Chloroethane	15.4-24.6
2-Chloroethylvinyl ether	12.0-28.0
Chloroform	15.0-25.0
Chloromethane	11.9-28.9
Dibromochloromethane	13.1-26.9
1,2-Dichlorobenzene	14.0-26.0
1,3-Dichlorobenzene	9.9-30.1
1,4-Dichlorobenzene	13.9-26.1
1,1-Dichloroethane	16.8-23.2
1,2-Dichloroethane	14.3-25.7
1,1-Dichloroethane	12.6-27.4
T-1,2-Dichloroethene	12.8-27.2
1,2-Dichloropropane	14.8-25.2
C-1,3-Dichloropropene	12.8-27.2
T-1,3-Dichloropropene	12.8-27.2
Methylene chloride	15.5-24.5
1,1,2,2-Tetrachloroethane	9.8-30.12
Tetrachloroethene	14.0-26.0
1,1,1-Tetrachloroethane	14.2-25.8
1,1,2-Trichloroethane	15.7-24.3
Trichloroethene	15.4-24.6
Trichlorofluoromethane	13.3-26.7
Vinyl chloride	13.7-26.3
Benzene	15.4-24.6
Chlorobenzene	16.1-23.9
1,2-Dichlorobenzene	13.6-26.4
1,3-Dichlorobenzene	14.5-25.5

*** Based upon 20 ug/l target

TABLE 13-10 cont

QC ACCEPTANCE CRITERIA FOR METHODS 601/602

PARAMETER	RANGE ug/l
1,4-Dichlorobenzene	13.9-26.1
Ethylbenzene	12.6-27.4
Toluene	15.5-24.5

TABLE 13-11

QC ACCEPTANCE CRITERIA FOR METHODS 8021B

PARAMETER	RANGE ug/l
Bromodichloromethane	77-113
Bromoform	86-116
Bromomethane	85-109
Carbon tetrachloride	83-101
Chlorobenzene	73-109
Chloroethane	84-108
2-Chloroethylvinyl ether	60-140
Chloroform	89-107
Chloromethane	69-123
Dibromochloromethane	81-117
1,2-Dichlorobenzene	85-109
1,3-Dichlorobenzene	83-119
1,4-Dichlorobenzene	85-127
1,1-Dichloroethane	82-118
1,2-Dichloroethane	88-112
1,1-Dichloroethane	82-118
T-1,2-Dichloroethene	87-111
1,2-Dichloropropane	79-105
C-1,3-Dichloropropene	76-124
T-1,3-Dichloropropene	76-124
Methylene chloride	88-106
1,1,2,2-Tetrachloroethane	64-136
1,1,1,2-Tetrachlorotehane	85-115
Tetrachloroethene	81-111
1,1,1-Tetrachloroethane	89-107
1,1,2-Trichloroethane	87-117
Trichloroethene	69-111
Trichlorofluoromethane	87-105
Vinyl chloride	77-113
Benzene	87-111
Ethylbenzene	84-114
Toluene	82-118

***Based upon a 100 ug/l target

TABLE 13-11 cont

QC ACCEPTANCE CRITERIA FOR METHODS 8021B

PARAMETER	RANGE ug/l
Bromochloromethane	87-105
Methyl-t-Butyl Ether	N/A
2,2-Dichloropropane	87-105
1,1-Dichloropropene	87-105
Dibromomethane	82-118
1,3-Dichloropropane	81-117
1,2-Dibromoethane	79-115
m,p-Xylene	79-115
o-Xylene	82-130
Styrene	88-112
1,2,3-Trichloropropane	87-111
Isopropylbenzene	84-120
Bromobenzene	76-118
Propylbenzene	78-120
2-Chlorotoluene	84-114
4-Chlorotoluene	75-117
1,3,5-Trimethylbenzene	89-113
t-Butylbenzene	89-107
1,2,4-Trimethylbenzene	75-117
sec-Butylbenzene	79-121
p-Isopropyltoluene	89-107
Butylbenzene	76-112
1,2-Dibromo-3-Chloropropane	62-122
1,2,4-Trichlorobenzene	43-139
1,2,3-Trichlorobenzene	75-129
Naphthalene	77-119
Hexachlorobutadiene	79-121

***Based upon a 100 ug/l target

14.0 PERFORMANCE AUDITS

14.1 SYSTEM AUDITS

System audits consist of a review and evaluation of all components of laboratory operation. This will involve the assessment of compliance with all regulatory agency regulations and adherence to laboratory QA guidelines and QC procedures. Once the evaluation is complete, the evaluation sheet is reviewed by the department supervisor and the Technical Director. The reports become a part of the permanent laboratory record.

14.1.1 Internal audits are performed semi-annually by the QA Officer. The checklist used during these audits is summarized in Table 14-1.

14.1.2 External Audits

The laboratory is inspected by many regulatory agencies (AIHA, Tennessee, Utah, California, Florida, New York, Arizona, West Virginia, North Carolina, Iowa, New Jersey, Wisconsin, Corps of Engineers). These inspections occur at different times during the year except the Corps of Engineers audits the laboratory every 18 months.

14.2 PERFORMANCE AUDITS

Performance audit samples are submitted semi-annually by the USEPA (WP and WS series). Performance audit samples are submitted on an unscheduled basis from the USACE. The State of New York submits audit samples two times per year; Proficiency Samples are also available from Wisconsin. Performance samples are also analyzed from Commercial Sources and these results may be used for Certification requirements in additional states.

Internal audit samples are submitted on a random basis with a minimum frequency of six months.

TABLE 14-1

INTERNAL AUDIT CHECKLIST

1.0 ORGANIZATION AND RESPONSIBILITY

1.1 Verify that the Laboratory Director is responsible for the following:

A. The design and implementation of the QA program

B. The design and maintenance of the organizational chart

C. Assisting with the review of the final reports

D. The maintenance of the LIMS database

1.2 Verify that the Quality Assurance Officer is responsible for the following:

A. Daily monitoring of quality control procedures

B. Initiating in-house proficiency evaluation samples

C. Keeping the Lab Manager and the Technical Director apprised of changes in regulations and any deficiencies noted in proficiency sample results or on-site audits.

D. Monitoring of the health and safety procedures of the laboratory

E. Monitoring and disposal of laboratory waste

F. Assisting in the review of final reports

1.3 Verify that the Technical Director is responsible for the following:

A. Maintaining the Standard Operating Procedures of the laboratory

B. Final review of analytical data

C. Monitoring all technical procedures to insure that proper procedures are followed

D. Assisting in the review of the final reports

1.4 Verify that the Technical Support Manager is responsible for the following:

A. The tracking and security of all labortory technical documents

B. The printing and delivery of final laboratory reports

C. Receiving of laboratory supplies

D. Daily LIMS maintenance under the direction of the Laboratory Manager

1.5 Verify that the sample log-in supervisor is responsible for the following:

A. Supervising the entry of data into the LIMS during sample repeipt

B. Notifying the technical areas when samples with accelerated turn around times are received.

C. Supervising the storage of samples from the time of acceptance until disposal

1.5 Verify that the technical supervisors are responsible for the following

A. Evaluating instruments, software, and personnel performance

B. Instrument maintenance

C. Supervision of all analytical procedures

D. Maintenance of QC records

E. Communicating with the Technical Director when problems arise that adversely affect the performance of the area

1.6 Verify that the analyst is responsible for the following"

A. Perfoming the requested analysis according to procedures found in the SOP

B. Reporting any noted anomalies directly to his supervisor

C. Performing routine maintenance on instrumentation

2.0 EDUCATION AND TRAINING OF PERSONNEL

2.1 Verify that personnel have been trained on QA procedures and are familar with laboratory SOP's.

2.2 Verify that there is documentation of approval for all analysts to perform analyses in the section.

2.3 Verify that the employee records have documentation of education and training

3.0 SAMPLE CUSTODY

3.1 Verify that samples received by the laboratory have a completed chain of custody with them.

3.2 Verify that samples are stored properly, e.g., refrigeration, VOA's segregated, etc.

3.3 Verify that all project folders have all data (or pointers to the data) and a copy of the chain of custody.

3.4 Verify that the LIMS security system restricts access depending upon clearance code.

4.0 ANALYTICAL PROCEDURES

4.1 Verify that the laboratoy SOP are updated regularly and are available in each section.

4.2 Verify that there are no unapproved methods in use in the areas.

4.3 Verify that MSDS are available in the sections.

4.4 Verify that all in-house reagents are dated and initialed by the person making the reagent.

4.5 Verify that all standards can be traced to the NBS or NIST through the use of a unique numbering system and that those numbers are recorded properly in log books.

4.6 Verify the documentation of the laboratory grade water.

4.7 Verify that laboratory glassware is cleaned according to approved protocol.

4.8 Verify that hazardous waste is properly stored, disposed of properly, and records are maintained.

5.0 INSTRUMENTATION CALIBRATION

5.1 Verify that each instrument type has a written protocol for periodic calibration.

5.2 Verify that a record of calibration exists for individual instruments.

6.0 PREVENTIVE MAINTENANCE

6.1 Verify that there is documentation of a continuing maintenance record for each instrument.

7.0 ASSESSMENT OF ACCURACY AND PRECISION

7.1 Verify that the QA Officer introduces blind QC chech samples into the analytical system routinely.

7.2 Verify that the analyst has access to acceptable ranges for all QC in the department.

7.3 Verify that all QC charts are pulled and reviewed monthly.

8.0 DATA REDUCTION, VALIDATION, AND REPORTING

8.1 Verify that the analyst computes the final result and that the LIMS results are compared to the manual result.

8.2 Verify that all required data review steps are being followed and that the initials of the person reviewing the data is present.

8.3 Verify that the final reports are reviewed and signed by the Laboratory Director, the Quality Assurance Officer, or the Technical Director.

9.0 CORRECTIVE ACTION

9.1 Verfiy that proper protocol is followed when changes are required to the sample database.

10.0 PERFORMANCE AUDITS

10.1 Verify that system audits are performed to assess compliance with all regulatory agencies and adherence to laboratory QA guidelines and and QC procedures.

10.2 Verify that internal audits are performed semi-annually by the QA Officer.

10.3 Verify that blind internal performance audit samples are analyzed semi-annually.

11.0 QUALITY ASSURANCE REPORTS

11.1 Verify that Qualtiy Assurance Reports are prepared quarterly by the QA Officer.

11.2 Verify that the QA Reports are reviewed and signed by the Laboratory Manger and the Technical Director.

15.0 QUALITY ASSURANCE REPORTS

Quality assurance reports will be prepared quarterly by the QA Officer. These reports will be reviewed by the Technical Director and the Laboratory Manager. All resulting actions will be documented and attached as an addendum to the report and returned to the QA Officer for filing.

The quality assurance report will include the following items:

A. Accuracy and precision assessment

Accuracy and precision will be assessed by evaluating computer generated recovery and duplicate precision data. The means and ranges will be compared to those outlined in the individual methods. Discrepancies will be noted and addressed accordingly. Trends in the data will be noted and addressed if present.

B. Method detection limits

Method detection limits are verified and updated annually. The verification process should be certified and the data resulting from the verification process should be compared to historical data a well as the data listed by the EPA in the appropriate manual.

C. System audits

The result of the most recent system audit should be included.

D. Performance audits

The result of the most recent blind performance audit sample set should be included with a listing of acceptable ranges. A review of any extemal performance audit sample results received since the last QC report should be listed.

Any QA/QC problems encountered since the last report should be summarized and included in the report. This will include a brief description of the problem and corrective action taken.

The report must have the signature of the Technical Director, the QA Officer, and the Laboratory Director before filing.

APPENDIX B

LABORATORY STANDARD OPERATING PROCEDURES

CHLORIDE (MERCURIC THIOCYANATE)

1.0 REFERENCE
- 1.1 EPA 600, Method 325.2
- 1.2 Standard Methods, 17th Ed., Method 4500 - Cl E.
- 1.3 SW-846 Method 9251

2.0 REAGENT
- 2.1 Stock mercuric thiocyanate solution - Dissolve 4.17 g mercuric thiocyanate in 1L of methanol.
- 2.2 Stock Ferric Nitrate reagent (0.5 M) - Dissolve 202 g ferric nitrate in 800 ml water. Add 25 ml conc nitric acid and dilute to 1 liter with DI water.
- 2.3 Combined Color Reagent - Mix 75 ml of stock mercuric thiocyanate solution with 75 ml of stock ferric nitrate reagent and dilute to 500 ml of DI water. Invert to mix. Vacuum filter through 0.45 um filter.
- 2.4 Chloride stock standard, 1000 ug/ml, commercial.

3.0 AUTOMATED PROCEDURE
- 3.1 Prepare following standards:
  100, 50, 25, 10, 5.0, 2.5, 1.0 and 0.0 ug/ml chloride.
- 3.2 Open chloride method from PC. (ref. Lachat Quik-Chem S.O.P. #39)
- 3.3 Build and load tray with standards, blanks, check standards and samples to be analyzed. Place standards in sampler in order of decreasing order. Start tray.
- 3.4 Prepare standard curve by plotting response vs. concentration. Successful calibration requires a correlation coefficient greater than 0.99
- 3.5 Verify calibration at the beginning, every 10 samples and at the end of the batch. The standard must be within 5% of the true value.
- 3.6 PQL for chloride is 1.0 mg/l.

4.0 QA/QC
- 4.1 Run a blank, spike and spike duplicate every 20 samples or batch if smaller.
- 4.2 Concentration in blank must be less than PQL. Spike recovery must be 80-120% with an RPD of < 20.
- 4.3 If concentration of sample exceeds calibration, dilute with DI water and reanalyze.
- 4.4 If particulates are observed centrifuge and decant prior to analysis..

CORROSIVITY

1.0 REFERENCE:
- 1.1 SW-846, Method 1110
- 1.2 SW-846, Method 9040A

2.0 MAINTENANCE: None required.

3.0 REAGENTS
- 3.1 Concentrated sulfuric acid, reagent grade, commercial.
- 3.2 Methanol, reagent grade, commercial.
- 3.3 pH Buffer (calibration), commercial.
- 3.4 Saturated calcium hydroxide solution (pH = 12.45)

4.0 PROCEDURE
- 4.1 Check pH of aqueous sample. If the pH of the sample is less than 2 or greater than 12.5, the sample is corrosive. NO FURTHER TESTING REQUIRED. Report results. Confirm alkaline pH by equilibating to 25 C. Check against saturated calcium hydroxide standard. Report results.
- 4.2 The coupons (SAE 1020 steel washers or strips) are scrubbed with a mild abrasive ie. steel wool, scotchbrite, etc. and rinsed with hot water and dried.
- 4.3 Weigh the washer on an analytical balance, record weight to nearest 0.0001 g.
- 4.4 Suspend the coupons in a vessel and add sufficient volumn of blank, control, or sample to obtain a 40 : 1 ratio of volumn to coupon area. If the sample is a solid fill the vessel about 1/3 full and add enough distilled water to form a slurry to ensure coupon is uniformly contacted with the sample maintaining the 40 : 1 ratio. Suspend coupon with a non-metallic support. Place teflon stirring bar in vessel. Cap or seal vessel.
- 4.5 Place vessel on a hot plate and heat to 55° C (130 degrees F) for 24 hours with constant stirring.
- 4.6 Remove coupons from vessel and gently scrub with mild abrasive, be careful not to remove any fresh metal.
- 4.7 Allow coupons to dry and reweigh. The difference between the initial and final weight is the weight loss used in the calculation.

5.0 CALCULATIONS
- 5.1 Coupon Surface Area Calculation:

  Area = (3.14 x r2 outside - 3.14 x r2 inside)(2) + (T x 3.14 x D) + (T x 3.14 x d)

where A = surface area
T = thickness
D = diameter of coupon
d = diameter of mounting hole
r2 = radius squared

All dimensions in centimeters.
Surface area for large coupons averages 22.4 sq.cm.
Surface area for small coupons averages 6.877 sq. cm.

NOTE: rectangular coupons may be used.

5.2 Corrosion Rate:

$$\text{millimeters/year} = \frac{\text{wt. loss x 11.145}}{\text{area x time}}$$

where: wt. loss is in milligrams, area is square centimeters, time is in hours
corrosion rate is millimeters/year

If the calculated rate is greater than 6.35 mm/yr the sample is corrosive.

6.0 QUALITY CONTROL

6.1 One blank consisting of distilled water is run per sample batch.
6.2 One control consisting of 50 : 50 of sulfuric acid / distilled water per sample batch.
6.3 Analyze duplicate samples on a routine basis.
6.4 Record temperature if pH > 12.0.
6.5 Calibrate electrode with saturated calcium hydroxide solution (pH = 12.45) at 25 C.

METHOD 9010/9012/335/CLP

CYANIDE, TOTAL, AMENABLE AND REACTIVE

1.0 SCOPE

1.1 This method is used to determine the concentration of inorganic cyanide in water and soils. Cyanide is released under acidic conditions and distilled to minimize interference and collected in a alkaline absorbing solution. The cyanide is then reacted with Chloramine-T at a pH < 8, the color is developed using pyridine-barbituric acid. All standards and samples must be at the same concentration of sodium hydroxide to obtain color of comparable intensity.

2.0 INTERFERENCES

2.1 Most interferences are eliminated by distillation.

2.2 Oxidizing agents like chlorine decompose cyanides. Excess chlorine is removed by treating with sodium arsenite.

2.3 Sulfide interferes with color development. For SW-846 method 9010/9012 remove by adding bismuth nitrate, for 335.2 CLP-M treat with cadmium carbonate before distillation.

2.4 Nitrate and nitrites may produce high results. Eliminate by adding sulfamic acid just before distillation. Do not use for 335.2 CLP-M.

3.0 REAGENTS (Common)

3.1 Sodium Hydroxide (1.25N) - Dissolve 50 g NaOH in one liter of DI water. Other concentrations may be made by dilutions.

3.2 Bismuth Nitrate (0.062M) - Dissolve 30 g bismuth nitrate in 100 ml DI water, add 250 ml of acetic acid, mix and dilute to one liter with DI water.

3.3 Sulfuric Acid (18N) - Carefully add 500 ml conc. H_2SO_4 to 500 ml DI water.

3.4 Magnesium Chloride (2.5M - Dissolve 500 g of MgCl in one liter of DI water.

3.5 Sulfamic acid (0.4N) - dissolve 40 g H2NSO3H in 1 L water

3.6 Sodium Arsenite, reagent grade, commercial - prepare a 0.1 N solution by adding 3.2 g to 250 ml DI water.

3.7 Cyanide Stock Solution - Dissolve 2.51 g of KCN and 2.0 g KOH in 900 ml DI water. Standardize against 0.0192N silver nitrate. Dilute to obtain concentration where 1.0 ml = 1000 ug CN. Purchased standards may be used in place of prepared solution. Dilute to make a 10 ppm working standard. Standardize against silver nitrate titrant weekly.

3.8 Silver nitrate (0.0192 N) - weigh 3.2647 g of dried AgNO3, dissolve in 1 L water.

3.9 Rhodanine indicator - dissolve 20 g p-dimethyl-amino-benzalrhodamine in 100 ml acetone.

METHOD 9010/9012/335/CLP

3.10 Cadmium carbonate, reagent grade, commercial source.

MANUAL

3.10 Sodium Dihydrogen Phosphate (1M) - Dissolve 13.8 g sodium phosphate in 100 ml DI water. Keep refrigerated.

3.11 Chloramine-T (0.44%) - Dissolve 1.0 g of white, water soluble chloramine-T in 100 ml DI water. Keep refrigerated.

3.12 Pyridine-Barbituric Acid - Place 6.0 g of barbituric acid in a container, wet with water, add 30.0 ml of pyridine and mix, add 6.0 ml of conc. HCl and mix, cool, dilute to 100 ml with DI water. Store in dark, cool location.

AUTOMATED

3.13 Sodium Hydroxide (eluent): 0.25 M - Dissolve 10 g NaOH in 1 L DI water.

3.14 Phosphate buffer 0.71 M - Dissolve 97 g anyhydrous potassium di - hydrogen phospate in 1 L DI water. Prepare fresh monthly.

3.15 Chloramine - T - Dissolve 2 g chloramine - T hydrate in 500 ml DI water. Prepare fresh daily.

3.16 Pyridine-Barbituric Acid - Place 15.0 g of barbituric acid in a container wet with water, add 75 ml pyridine and mix, add 15 ml HCl and mix. Cool. Dilute to 250 ml with DI water. Prepare fresh weekly.

4.0 PROCEDURE

4.1 Pretreatment for Cyanides Amenable to Chlorination

4.1.1 This test must be performed under amber light to prevent decomposition of K3(FeCN6).

4.1.2 For midi-distillation take a 50 ml aliquot of sample and add dropwise a solution of 0.35 M Calcium Hypochlorite (5g to 100 ml DI water) while stirring. Maintain the pH between 11 to 12 with 1.25N NaOH. Confirm the presence of excess chlorine by using moistened KI-starch indicator paper ie. a blue color is positive.

4.1.3 Continue stirring and periodically confirming the presence of excess chlorine for one hour, add 0.35M calcium hypochlorite if needed. After one hour add 0.1N sodium arseite until KI/starch paper no longer tums blue. Add 1.0 ml excess 0.1N sodium arsenite.

4.1.4 Test as below for total cyanide. The difference between the total cyanide and the cyanide value in the above treated sample is the cyanide ameniable to chlorination.

4.2 Extraction Procedure for Solids and Oily Waste

4.2.1 If the waste contains solids or oily mater that prohibits homogenization in

METHOD 9010/9012/335/CLP

the fritter bubbler used for distillation the sample should be extracted in a solution of DI water to which the pH is > 10. Preferrably 25 g is extracted with 500 ml fluid (10:1 alkaline water to hexane). Extract for 16 hours. Adjust sample weight to extract volume to achieve standard soil detection levels. Proceed with distillation sequence. Also referred to as soluble cyanides.

4.3 Distillation (Macro)for Reactivity:

4.3.1 Prior to distillation check for chlorine using KI-starch paper, if blue, add a few milliliters of sodium arsenite until no color change then an extra 5.0 ml/L of sample.

4.3.2 Place 10 g of sample diluted to 250 ml in a one liter boiling flask, add 50.0 ml of 0.25 N NaOH to scrubber, connect nitrogen gas to macro setup. Turn on, set flow to 60 ml, allow to purge for 1 to 2 minutes.

4.3.3 Add 250 ml of 0.2 N H_2SO_4 to addition funnel, open stopcock and add to boiling flask for final conc. of 0.1 N.

4.3.4 Start timer, begin stirring solution being sure not to create a vortex, after 30 minutes close off nitrogen and determine concentration of cyanide per p. 4.7..

4.4 Distillation (Midi) for 9010/9012:

4.4.1 Test for chlorine and sulfide per p. 4.3.1.

4.4.2 Pipet 50.0 ml of sample or aliquot diluted to 50.0 ml or 1.0 g with 50.0 ml DI water into Reflux tube. Add 0.2 g sulfamic acid. Mix for 3 minutes.

4.4.3 Add 50.0 ml of 0.25 N NaOH into Absorber tube. If sulfide is suspected add 5 ml bismuth nitrate solution.

4.3.4 Connect tubing, turn on chiller and vacuum pump, adjust flow controllers to obtain about 3 bubbles per second.

4.4.5 Add 5.0 ml of 50% H_2SO_4 through air inlet of each Reflux tube (pH < 2.0). Confirm pH.

4.4.6 Add 2 ml $MgCl_2$ solution through air inlet of Reflux tube.

4.4.7 Turn "ON" heating elements, set timer for 60 minutes.

4.4.8 Allow to cool for 15 minutes, turn "OFF" vacuum pump and chiller.

4.4.9 Transfer solution in Absorber tube to labelled container, adjust volume to 50.0 ml with DI water.

4.5 Distillation (Midi) for 335.2 CLP-M:

4.5.1 Test for chlorine per p. 4.3.1. Test for sulfide using lead acetate paper, if positive, treat 200 ml of sample with powdered cadmium carbonate. A yellow precipitate indicates sufide, continue treating until the lead acetate

METHOD 9010/9012/335/CLP

test is negative. Filter the treated sample through dry filter paper. Avoid a large excess of cadmiun carbonate.

4.5.2 Pipet 50.0 ml of sample or 1.0 g soil diluted to 50.0 ml with DI water into the distillation tube, add 2 or three boiling chips, place tube in heater block.

4.5.3 Add 50.0 ml of 0.25 N NaOH to absorbing tube, connect distillation device.

4.5.4 Turn vacuum on and adjust each needle valve to obtain a bubbling rate of about 3 per second. After about 5 minutes slowly add 5 ml of 50 % sulfuric acid through the distillation head, allow to mix for about 5 minutes, confirm that pH is less than 2. Add 2 ml of magnesium chloride solution, if excess foaming is observed then add 2ml more of magnesium chloride solution.

4.5.5 Turn on heating block, set timer for 120 minutes, check bubble rate periodically and adjust as needed to maintain about 3 bubbles per minute.

4.5.6 After cooling, carefully disassemble absorbing tube, adjust volume to 50.0 ml with 0.25 N NaOH if needed, transfer to plastic centrifuge tube, cap, store at 4 C until analysis. Analysis must be within 12 days.

4.6 Spectrophotometry (Manual)

4.6.1 Daily prepare 7 calibration standards and a blank by diluting stock CN solution as follows:

ml 10 ppm std	final vol (ml)	conc. (ug/ml)
0.0	10	0
0.005	10	0.005
0.01	10	0.01
0.02	10	0.02
0.05	10	0.05
0.1	10	0.1
0.2	10	0.2
0.4	10	0.4

Total volume of each standard and sample must be identical.

4.6.2 Using 5 ml sample, place in test tube and add 3.0 ml of sodium dihydrogen phosphate solution, 0.5 ml chloramine-T, mix and immediately add 1.0 ml pyridine-barbituric acid. Mix well.

4.6.3 Allow to react for at least 8 minutes. Read absorbance at 578 nm within

METHOD 9010/9012/335/CLP

15 minutes. Calculate concentration per p. 4.7.6.

4.6.4 Prepare standard curve by plotting absorbance vs. CN concentration.

4.6.5 If concentration exceeds highest standard dilute and re-analyze.

4.7 Spectrophotometry (Automated)

4.7.1 Daily prepare 7 calibration standards 0.005, 0.01, 0.02, 0.05, 0.1, 0.2 and 0.4 ug/ml and a blank by diluting working CN solution.

4.7.2 Place standards, QC samples, and samples on autosampler.

4.7.3 Open cyanide method from PC, open new tray and build tray by entering sample I.D. and corresponding cup number. (Ref. Lachat S.O.P.)

4.7.4 Run tray by clicking on run tray icon. Standards will be run and an analytical curve will be calculated from peak areas. Samples will then be calculated from curve and reported in concentration.

4.7.5 If concentration exceeds highest standard dilute and re-analyze.

4.7.6 Calculate concentration as follows:

$$\text{CN (mg/L or mg/kg)} = \frac{\text{(conc. from cal. curve)(dilution)(final vol. in ml)}}{\text{(sample vol. in ml or sample wt. in g)}}$$

5.0 QUALITY CONTROL

5.1 Analyze a distilled check standard, low and high concentration, from a second source with every batch. Recovery must be within 10% of non-distilled standards.

5.2 A blank and matrix spike (0.04 mg/L) must be run for every batch or 10 samples. Recovery must be within lab limits. The distilled blank must be less than the PQL.

5.3 For samples containing sulfides calibration standards must be distilled. This is not required for 335.2 CLP-M.

5.4 Analyze one duplicate sample per batch or every 10 samples, %D < 20 or reanalyze samples.

5.5 Analyze a mid-level (non-distilled) check standard, from a second source, every 10 samples and at end of batch. %D < 15 or reanalyze sample.

5.6 Samples must be collected in glass or plastic. Aqueous samples must be preserved with NaOH to a ph > 12. Cool all to 4 C. Extract/distill within 14 days. Analyze distillate within 12 days for CLP-M 335.2.

METHOD 9010/9012/335/CLP

6.0 REFERENCE

6.1 SW-846, Method 9010
6.2 EPA 600, Method 335.2
6.3 EPA 600, Method 335.3
6.4 SW-846, Method 9013
6.5 SW-846, Method 9012
6.6 CLP-M ILM04.0 Method 335.2
6.7 SW-846 Chapter 7

DIGESTION - HOT PLATE

1.0 REFERENCE

1.1 SW-846 Method 3005 (Total Recoverable for Al,As,Ba,Be,Cd,Ca,Cr,Co,Cu,Fe,Pb,Mg,Mn,Mo,Ni,K,Se,Ag,Na,Tl,V and Zn)

1.2 SW-846 Method 3010 (Total for Al,As,Ba,Be,Cd,Ca,Cr,Co,Cu,Fe,Pb,Mg,Mn,Mo,Ni,K,Se,Na,Tl,V and Zn)

1.2 SW-846 Method 3050 for Al,As,Ba,Be,Cd,Ca,Cr,Co,Cu,Fe,Pb,Mg,Mn,Mo,Ni,Os,K,Ag,Na,Se,Tl,V and Zn

1.3 Contract Laboratory Program, SOW No. ILM0 4.0 for Al,Sb,As,Ba,Be,Cd,Ca,Cr,Co,Cu,Fe,Pb,Mg,Mn,Ni,K,Se,Ag,Na,Tl,V and Zn.

1.4 EPA 600, Method 200.7

1.5 Standard Methods, Method 3030 C

2.0 REAGENTS

2.1 Water Spike (all metals except silver) add:

10.0 ml Al	1.25 ml Cu	2.5 ml Zn
10.0 ml Ba	5.0 ml Fe	2.5 ml Mo
0.25 ml Be	0.25 ml Pb	0.25 ml Se
1.0 ml Cd	2.5 ml Mn	0.25 ml As
1.0 ml Cr	2.5 ml Ni	0.25 ml Tl
2.5 ml Co	2.5 ml V	0.50 ml Sb

of 1000 ppm stock standards to a 100 ml volumetric flask and bring up to volume with 1% nitric acid.
-- Add 1 ml of this solution to water samples to be spiked.

2.2 Soil Spike (all methods except silver) add:

10.0 ml Al	1.25 ml Cu	2.5 ml Zn
10.0 ml Ba	5.0 ml Fe	2.5 ml Mo
0.25 ml Be	2.5 ml Pb	0.5 ml As
0.5 ml Cd	2.5 ml Mn	0.5 ml Se
1.0 ml Cr	2.5 ml Ni	0.5 ml Tl
2.5 ml Co	2.5 ml V	2.5 ml Sb

of 1000 ppm stock standards to a 100 ml volumetric flask and bring up to volume

with 1% nitric acid.
-- Add 2 ml of this solution to soil samples to be spiked.

2.3 Silver Spike - Add 0.25 ml 1000 ppm stock standard to 100 ml volumetric flask and bring up to volume with 1% nitric acid.
-- Add 1 ml of this solution to water sample to be spiked.
-- Add 2 ml of this solution to soil samples to be spiked.

2.4 TCLP Spike - Add 5 ml As and Se (1000 ppm ea.) and 25 ml Pb and Cr (1000 ppm ea.) to a 100 ml volumetric flask and bring up to volume with 1% nitric acid.
-- to TCLP sample add 1 ml of TCLP spike solution, 1 ml stock Barium (1000 ppm) solution and 50 ul of silver stock solution (1000 ppm.).

3.0 PROCEDURE

3.1 Water (Method 200.7 and 3005 for Total Recoverable)

3.1.1 Transfer 50.0 ml of well mixed sample to a beaker, add 1.0 ml conc. HNO3 and 2.5 ml conc. Hcl.

3.1.2 Cover with a watch glass, place on a hot-plate adjusted to 85 C, reduce volume to about 15 ml (DO NOT BOIL).

3.1.3 Remove fro heat, allow to cool, dilute with DI to 50.0 ml.

3.2 Water (Method 3010 for Total Metals except Silver and Antimony)

3.2.1 Shake sample and transfer 50 ml to a clean, acid washed 100 ml beaker. Add 1.5 ml of conc. nitric acid.

3.2.2 Cover with a watch glass and heat to approximately 95° C, reduce volume to about 10 ml. Do not allow the samples to boil or go to dryness. Allow the samples to cool, add 3 ml conc. HNO3, heat with additions of acid until digestate is light in color, reduce volume to about 5 ml but do not allow to go dry, cool, add 5.0 ml 1:1 Hcl, cover and reflux for 15 minutes

3.2.3 Adjust the final volume to 50.0 ml with DI water using volumetric glassware and transfer to a 50 ml polypropylene tube until analysis.

3.3 Water (CLP ILMO 4.0)

3.3.1 Shake sample, transfer 50 ml to beaker, add 1.0 ml of 1:1 HNO3 and 5.0 ml 1:1 Hcl, cover with watch glass and heat at 92 C to 95 C for two hours or until volume is about 15 to 20 ml. Do not allow to boil.

3.3.2 Cool, filter, adjust volume to 50.0 ml with DI.

3.4 Water (3030 C)

3.4.1 Transfer 100 ml of sample to 150 ml beaker, add 5 ml of 1:1 HCl, heat on hotplate at 95 C for 15 minutes.

3.4.2 Cool, filter through 0.45 um membrane filter, adjust final volune to 100 ml with DI. Extract is ready for analysis.

3.5 Sediment, Sludges, Soils and Feeds (Method 3050 and CLP ILMO 4.0)

3.5.1 Accurately weigh approximately 1 gram of sample into a 100 ml beaker. If the sample is not homogeneous, a representative portion should be taken.

3.5.2 Add 10 ml of 1:1 nitric acid, mix and cover with a watch glass. Reflux the sample at approximately 95° for 10 to 15 minutes without boiling.

3.5.3 Allow the sample to cool and add 5 ml of concentrated nitric acid. Replace the watch glass and reflux for 30 minutes. Do not allow the volume to drop below 5 ml while maintaining a covering of the beaker bottom.

3.5.4 Allow the sample to cool and add 2 ml of DI water and 3 ml of 30% hydrogen peroxide. Cover with watch glass and heat the sample until the effervescence subsides and allow to cool.

3.5.5 Continue to add peroxide in 1 ml aliquots until the effervescence is minimal and the appearance of the sample does not change. No more than 10 ml of peroxide should be added.

3.5.6 Add 5 ml of concentrated HCl and 10 ml of water, return the covered beaker to the hot plate and reflux for an additional 15 minutes without boiling. Cool and dilute to 100.0 ml with DI. Remove particulate prior to analysis by filtering through No. 41 Whatman or equivalent.
NOTE: do not use Hcl for feeds or for samples analyzed by GFAA

4.0 QUALITY CONTROL

4.1 A blank should be digested with each batch of no more than 20 samples.

4.2 One sample of each batch should be spiked along with a spike duplicate.

4.3 All final dilutions should be in class A glassware.

4.4 All glassware must be acid cleaned and DI rinsed prior to each use.

DIGESTION - MICROWAVE

1.0 SCOPE AND APPLICATION

This digestion procedure is suitable for water samples, mobility-procedure extracts, wastes that contain suspeded solids and soils for analysis by FLAA, GFAA and ICP. The procedure uses a hot-acid leach for determining the following metals: SW-846:Aluminum, Antimony, Arsenic (non FLAA), Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium (non FLAA), Silver if <0.1 mg/L, Sodium, Thallium, Approved for the following NPDES metals by ICP: Aluminum, Antimony, Arsenic, Barium if <0.5 mg/L, Cadmium if <0.5 mg/L, Chromium, Copper, Iron, Lead if <0.4 g/L, Manganese, Nickel, Selenium and Zinc.

2.0 SUMMARY OF METHOD

A closed digestion vessel heated by microwave radiation is used. 45 ml of aqueous sample plus 5 ml nitric acid or 0.5 g of soil plus 10 ml nitric acid is used for digestion.

3.0 INTERFERENCES

3.1 High levels of organics may require dilution to prevent over pressurizing the vessel.

4.0 INSTRUMENTATION

4.1 Microwave, commercial analytical, 1200 W nominal, CEM.

5.0 REAGERNTS

5.1 Nitric Acid, conc., reagent grade, commercial.

5.2 Hydrochloric Acid, conc., reagent grade, commercial.
Note: reagent blank and method blank must be less than MDL's.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Collect in clean, metal free, plastic containers.

6.2 Aqeous samples must be acidified to a pH < 2 with nitric acid.

7.0 PROCEDURE

7.1 Clean teflon liners using dilute nitric acid followed by DI rinse. Note: high concentrations may require more aggressive cleaning using hot 1:1 HCl followed by hot 1:1 HNO, and DI rinse.

7.2 Dry liners and place in microwave reaction vessel.

7.3 Transfer sample, 45.0 ml if water or 0.5 g is solid, and add acid as follows:

NPDES 3 ml 70% HNO3, 2 ml 37% HCl

SW-846 5 ml conc. HNO3 for water 10 ml conc. HNO3 for solids

Weigh each liner and record.

7.4 Place teflon pressure-ported top and reaction vessel cap each with new rupture membrane on vessel and tighten lightly (finger tight).

NOTE: Place pressure sensor top on most contaminated sample.

7.6 Place sealed vessels in carousel. Attach vent tubes to center collection vessel and pressure sensing line to transparent tube valve. Open valve. Fully seat carousel in microwave. Blanks must be used to fill carousel and balance power input.

7.7 Recall appropriate method from menu (F3). Load (F1) method.

NOTE: program per manufactuers recommendation to achieve the following conditions: Water - to 160 C =/- 4 C in 10 min, 165-170 C for 10 min.

Soil - to 175 C in less than 5.5 min, 170-180 C for remaining 10 minutes.

7.8 Start method (F4).

7.9 Record final pressure reached on batch sheet. Confirm correct temperatures achieved.

7.10 Close transparent valve and detach pressure sensing line on side farthest from reaction vessel.

7.11 Remove carousel and allow to cool for about 10 minutes.

7.12 Slowly open pressure valves on all vessels. Use caution.

7.13 Remove caps. Reweigh liner, record, if change in weight > 10% redigest sample.

7.14 Adjust soils to final volume with DI water.

8.0 QUALITY CONTROL

8.1 Each vessel requires new rupture membrane for each digestion.

8.2 Repeat digestion if membrane ruptures, alarm will sound, or if weight change exceeds 10 %.

8.3 Run a blank, spike, and duplicate with every batch.

8.4 Soak teflon liners in dilute nitric acid overnight.

8.5 Wear safety equipment when venting reaction vessels.

8.6 Use extreme caution when digesting organics. See supervisor.

8.7 Calibrate semi-annually microwave energy using the manufacturers programmed procedure. Power (watts) = (ΔT oC) (34.86). Calibrate with a full carousel.

9.0 REFERENCES

9.1 SW-846 Method 3015 Rev. 0, Sept. 1994

9.2 SW-846 Method 3051 Rev. 0, Sept. 1994

9.3 NPDES 40 CFR Part 136

METHOD 8270

EXTRACTABLE ORGANIC ANALYSIS (SEMI-VOLATILE) GC/MS

1.0 SCOPE AND APPLICATION

This procedure is used to determine the concentration of semi-volatile organics in aqueous and various liquid and solid wastes by GC/MS. It is applicable to neutral, acidic and basic compounds soluble in methylene chloride and able to be chromatographed.

2.0 SUMMARY OF METHOD

Samples are extracted with methylene chloride, concentrated and analyzed using capillary columns. Selected ions are quantitated with by the internal standard method. This method is suitable for most neutral, acidic and basic extractable organics.

3.0 INTERFERENCES

3.1 Any organics soluble in MeCl2 may interfere. Use appropriate cleanup to minimize interferences.

3.2 Contamination due to carryover can occur if high concentrations are found. Clean/replace injector liner or clip column, check with blanks, repeat samples if necessary.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph/Mass Spectrometer - HP-MSD, gas chromatographs are HP-5890-II.

4.2 Column: RTX-5 MS capillary, 30 m x 0.25 mm, 0.25 um film or equivalent.

5.0 REAGENTS

5.1 Methylene Chloride - Pesticide grade or equivalent, commercial source.

5.2 Stock Standard Solutions - Stock standard solutions may be prepared on a weight/volume basis using pure standard material, or may be purchased directly as certified solutions, i.e. Ultra, Supelco, etc. Store at -10 C. Good for 1 year.

5.3 Working Standard Solutions - Working standard solutions should be diluted from the stock standard solutions to give a working standard solution at concentrations of 2.0, 10.0, 20.0, 50.0, 80.0 and 100 ug/ml. The internal standard solution should be constant at 40 ug/ml for quantitation purposes in 20 % carbon disulfide. Store at 4 C. Good for 1 week.

5.4 Synthetic Soil, Sea Sand, precleaned, commercial source.

5.5 Internal Standard Solution – the following are used: 1,4-dichlorobenzene-d4, napthalene-d8, acenapthene-d10, phenanthrene-d10, chrysene-d12 and perylene-d12. Prepare at 4000 ug/ml by using 0.2 g each diluted to 50 ml with MeCl2 in 20

METHOD 8270

% CS2. Use 10 ul per 1.0 ml extract for a 40 ng/ul standard. Store at –10 C or less.

5.6 Tuning Standard – prepare a 50 ug/ml mix containing DFTPP, 4,4-DDT, pentachlorophenol and benzidine. Store at –10 C or less.

5.7 Surrogate Standard – the following are used: phenol-d6, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d5, 2-fluorobiphenyl and p-terphenyl-d14. Prepare each at 100 ug/ml in acetone. Use 1.0 ml per sample added prior to extraction.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Collect aqueous samples in 1L amber glass containers with a teflon-lined screw cap. Refrigerate. Extract within 7 days. Analyze extract within 40 days. Store extract at –10 C in the dark.

6.2 Collect soil/solid samples in a 4 or 8 oz. glass jar with teflon-lined screw cap. Refrigerate. Extract within 14 days. Analyze extract within 40 days. Store extract at –10 C in the dark.

7.0 PROCEDURES

7.1 Tuning - The GC/MS system must be tuned every 12 hours to meet the established criteria for ion abundance of decafluorotriphenylphosphine (DFTPP), DDT breakdown, response and tailing for benzidine and pentachlorophenol.

MASS	ION ABUNDANCE CRITERIA
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

Load the GC with the tune standard solution and the appropriate descriptor to meet the following conditions:
Initial temp. = 200 C for 1 min., ramp at 20 C/min to 250 C for 3 min.
Inject 1.0 ul (50 ng) tuning standard and begin data acquisition. Display the

METHOD 8270

chromatogram at the conclusion of the acquisition and list the mass data for the spectrum of DFTPP. DDT breakdown to DDD and DDE must be less than 20 % and benzidine and pentachlorophenol response should be normal with no visible tailing. If all of the specified criteria are met, generate a hardcopy of the spectrum, the mass abundance data and the parameters under which the scans were acquired. This data is filed in the QC file for documentation. If the relative abundances of the mass peaks do not conform to the required criteria, enter the tuning program of the data system and retune using autotune or manually using the reference compound FC-43. Repeat the tuning procedure. If the system is still not performing to the required specifications, corrective actions such as cleaning the source and analyzer, replace liner, clip column or re-calibrating the mass range is required.

METHOD 8270

7.2 Initial Calibration - At least a five point calibration curve for all analytes of interest is required before sample analyses can begin. Program the GC to run the following:
Initial temp. = 45 C for 1 min., initial rate at 30 C/min., temp. 2 = 160 C for 0 min., rate 2 = 8.5 C/min. to 320 C for 4 min.

Analyze each of the standards and generate an appropriate quantitation report of each analysis. Calculate the mean RF and RSD for each target. The RSD should be less than or equal to 15 for each target. If the RSD is greater than 15 linear regression is needed, the corellation coefficient must be at least 0.99. The RSD for each CCC must be less than or equal to 30. The 13 CCC compounds used are shown below:

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
diphenylamine	Phenol
Di-n-octylphthalate	Pentachlorphenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

Calculate the RF for the 4 SPCC compounds: N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. The RF must be greater than or equal to 0.05. Evaluate RT, the RRT of each target should be within 0.06 RRT units. If the initial calibration fails to meet the above criteria, corrective steps should be taken and documented in the maintenance manual and the calibration procedure repeated.

7.3 Daily Calibration - After the initial calibration has been successfully completed, a daily calibration standard should be analyzed at a concentration of 50 ug/ml every shift (12 hours). Set the GC to run the temperature program previously described and inject 1.0 ul of the 50 ug/ml standard. After the acquisition has completed, quantitate the standard and generate the response factors for each analyte. The response factors for the 4 SPCC compounds must be at least 0.05, the % RSD for the 13 CCC compounds must less than or equal to 20. If these requirements are not met, repeat the analysis of the daily calibration standard. If the values for the check compounds are still outside the regulatory limits, a new calibration curve must be established.

METHOD 8270

7.4 Sample Analysis

7.4.1 Sample Data Acquisition - After the relevant performance criteria have been completed and accepted, sample analysis can begin. Analyze 1.0 ul of the sample extract after adding 10 ul of internal standard solution. Acquire the mass spectral data and generate a quantitation report for each sample.

7.4.2 Data Analysis - The recovery and RT of the internal standards is checked to determine if the RT is within 30 seconds and recovery within –50 % to +100 % and that the surrogate standards are within historical limits. If any of the standards are outside the acceptable limits, reanalyze the sample. If the values remain outside the QC limits, corrective action must be taken. If the recovery of all standards is within the specified range, the concentration of each analyte detected is examined to ensure that they are within the calibration range of the instrument as established by the initial calibration curve. If any analyte is outside the calibration range, appropriate dilutions should be analyzed to bring these values into the working range of the instrument (i.e., <100 ug/ml).

8.0 QUALITY CONTROL

8.1 Method Blank - A method blank should be extracted with every batch of samples, not to exceed 20 samples per batch. The method blank should be analyzed prior to the reporting of any sample data to confirm that the system is free from any contamination. The method blank should not contain any analyte of interest at a level greater than the PQL for that analyte. If any analyte is detected at a level exceeding the PQL, re-analyze the blank.. If the contamination is persistent, all samples in the batch must be re-extracted.

8.2 Surrogate Recovery - The limits for surrogate recovery (%) are updated quarterly (see QA manual for specific limits). If any surrogate falls outside the required recovery limits, the sample must be re-extracted and re-analyzed. If the surrogates are still outside limits, the data should be flagged.

8.4 Matrix Spike/Spike Duplicate - a matrix spike and matrix spike duplicate should be extracted with every batch of samples. The percent recovery and RPD is compared to the acceptance criteria (LIMS historical). If any analyte fails this criteria, a QC check sample or LCS containing that analyte should be checked. If the recovery of the QC check standard is within acceptable levels, the data for the associate samples should be reported with notation of the failed matrix spike. If the recovery of the QC check standard does not meet the required limits, find and correct the problem and re-analyze all of the associated samples.

METHOD 8270

8.5 Calculations

8.5.1 Response factor = (area unk. x conc. IS) / (area IS x conc. unk.)

8.5.2 Mean RF = sum of RF of each calib. level / number of calib. levels
RSD = (std. dev.)(100) / mean RF

8.5.3 % Difference = (mean RF - RF of cal. verif. std.)(100) / mean RF

8.5.4 Final concentration, ug/ml or ug/g =
(quant. value)(dilution)(extract vol, ml) / initial vol., ml or g

9.0 REFERENCES

9.1 USEPA SW846, Method 8270 C, Rev. 3, Dec. 1996

EXTRACTION, BNA/PEST/PCB (SOIL)

1.0 REFERENCE
- 1.1 SW-846, Method 3550A
- 1.2 SW-846, Method 3620
- 1.3 SW-846, Method 3540 (Required for Organophosphorus Pesticides)

2.0 REAGENTS
- 2.1 Sodium sulfate, ACS grade, commercial.
- 2.2 Methylene chloride, pesticide grade, commercial
- 2.3 Acetone, pesticide grade, commercial.
- 2.4 Hexane, pesticide grade, commercial.
- 2.5 Alumina, neutral, commercial.
- 2.6 Acetonitrile (MeCN), HPLC grade, commercial source.

3.0 PROCEDURE (3550 - Sonication)
- 3.1 Weigh accurately about 30 grams of soil into a 400 ml beaker, dry the sample by adding 60 g sodium sulfate. Mix the sample well to a free-flowing mixture.
- 3.2 Add the appropriate surrogate or spike and add about 100 ml of MeCl2.
- 3.3 Submerge the beaker down to the solvent line in ice water.
- 3.4 Tune the horn, place probe tip 1/2 inch below solvent surface and sonicate the sample at a setting of 10 for 3 minutes using 50% pulse cycle. Decant the sample through a funnel containing sodium sulfate into a flask if layers are detected.
- 3.5 Repeat extraction two more times and pour the extracts into a K-D with 3-ball Snyder column apparatus for concentration.
- 3.6 Concentrate the extracts to less than 10 ml, rinse K-D with 10-20 ml $MeCl_2$ then bring to a final volume of 10.0 ml with $MeCl_2$.
- 3.7 **OPTIONAL** - For pesticide/PCB's remove 1.0 ml of the concentrate, add 5 ml of hexane, and concentrate to 1.0 ml using a micro Snyder column.
  - 3.7.1 Wet Florisil column by passing 1 ml hexane/acetone (8:2), add extract to the top of a column containing 2.5 g of Florisil. Elute with 10 ml hexane/acetone. Concentrate to 1.0 ml using a micro Snyder column.
    NOTE: Do not allow Florisil to go dry during clean-up.
    NOTE: For PBC only elution with hexane only is acceptable.
- 3.8 The remainder of the concentrate from step 3.6 is concentrated to 1.0 ml and transferred to a screw capped vial (BNA fraction).
  NOTE: If BNA is not requested, archive 9.0 ml concentrate.
- 3.9 For Pah only extract at ph=7, if samples are being prepped for HPLC solvent

exchange to acetonitrile. Exchange as follows:

3.9.1 Momentarily remove snyder and quickly add 4 ml MeCN, a new boiling chip, reattach snyder and over next 15-20 minutes reduce to app. 0.5 ml, remove K-D and cool.

3.9.2 Remove snyder and rinse with app. 0.2 ml MeCN, adjust final volume to 1.0 ml with MeCN. Transfer to 1.8 ml vial and cap.

4.0 PROCEDURE (3540 – Soxhlet)

4.1 Discard any water layer and any sticks, leaves, rocks, etc. Reduce by grinding to less than 1 mm diameter. Add 10 g anhydrous sodium sulfate to 10.0 g sample to obtain a free flowing mix. Transfer to extraction thimble.

4.2 Add 1.0 ml surrogate and spike if required and assemble extractor.

4.3 Place about 300 ml of 1:1 MeCl2:acetone in round bottom flask. Turn ON chiller, adjust temperature to obtain 4-6 cycles per hour, extract for 16 to 24 hours.

4.4 Turn OFF and allow to cool. Dry extract by passing through 10 cm column of anhydrous sodium sulfate, rinse and transfer extract to K-D, rinse extractor with extraction solvent, add to K-D. Concentrate to final volume of 10.0 ml.

4.5 Do not perform any additional clean-up, dilute if precipitate forms. Transfer extract to vial, cap with teflon-lined cap, store in dark at 4 C.

5.0 QUALITY CONTROL

5.1 A blank is extracted with each batch or 20 samples.

5.2 A matrix spike and matrix spike duplicate is extracted with each batch or 20 samples.

5.3 When preparing a spike/duplicate on a soil sample prepare a LCS using synthetic soil.

5.4 Store final extracts in amber vials at 4 C.

EXTRACTION, BNA, WATER

1.0 REFERENCE
- 1.1 SW-846, Method 3510

2.0 REAGENTS
- 2.1 Methylene chloride, ACS grade, commercial.
- 2.2 Sodium sulfate, ACS grade, commercial.
- 2.3 Sodium hydroxide (10N) - 40g diluted to 100 ml DI water.
- 2.4 Sulfuric acid (18N) - 50 ml acid to 50 ml DI water.
- 2.5 Acetonitrile (MeCN), HPLC grade, commercial source.

3.0 PROCEDURE
- 3.1 Mark liquid volume in sample container, shake, transfer to a 2 L separatory funnel. Adjust pH > 11 with sodium hydroxide or to < 2.
- 3.2 Add surrogate or spike. Rinse sample container with 60 ml of methylene chloride and transfer to the separatory funnel.
- 3.3 Measure original sample volume by filling to mark with DI water and measuring volume in graduated cylinder. Record.
- 3.4 Shake separatory funnel for 1-2 minutes with venting. Allow the layers to separate for at least 10 minutes.
- 3.5 Drain the solvent layer through a funnel containing sodium sulfate into a flask. Repeat extraction two more times and combine extracts.
- 3.6 Adjust the pH to < 2 with sulfuric acid or >11 with NaOH and repeat extractions.
- 3.7 Combine all extracts and concentrate using a K-D apparatus with 3-ball Snyder column to less than 1.0 ml. Rinse K-D with 5-10 ml $MeCl_2$ and attach a micro Synder column and concentrate to less than 1.0 ml. Adjust the final volume to 1.0 ml with $MeCl_2$ and transfer to an amber screw cap vial for storage.
- 3.8 For Pah only extract at ph=7, if samples are being prepped for HPLC solvent exchange to acetonitrile. Exchange as follows:
  - 3.8.1 Momentarily remove snyder and quickly add 4 ml MeCN, a new boiling chip, reattach snyder and over next 15-20 minutes reduce to app. 0.5 ml, remove K-D and cool.
  - 3.8.2 Remove snyder and rinse with app. 0.2 ml MeCN, adjust final volume to 1.0 ml with MeCN. Transfer to 1.8 ml vial and cap.

4.0 QUALITY CONTROL
- 4.1 Extract one blank for each batch or 20 samples per matrix.
- 4.2 Extract one matrix spike and matrix spike duplicate for each batch or 20 samples per matrix.

4.3 If a precipitate or phase separation occurs after cool-down, re-dilute to 5.0 ml.

4.4 Adjust temperature of water bath to obtain concentration from 10-20 minutes. Do not exceed.

4.5 During extraction if turbidity is detected the addition of 2 ml of 0.1 M EDTA may improve recovery by chelating with metal cations.

4.6 When preparing a spike/duplicate on a water sample prepare a LCS using in-house DI water.

EXTRACTION, PCB (FLUFF/WIPES/OILS)

1.0 REFERENCE
- 1.1 SW-846, Modified 3550
- 1.2 SW-846 Method 3620

2.0 REAGENTS
- 2.1 Hexane, Pesticide grade, commercial.
- 2.2 Sulfuric Acid, conc., reagent grade, commercial.
- 2.3 Florisil, activated at 1250 F, commercial source.

3.0 PROCEDURE
- 3.1 Fluff
  - 3.1.1 Place 50.0 g of sample in wide-mouth jar and add 125 ml hexane. Add surrogate. Cap.
  - 3.1.2 Place in mechanical shaker for 30 minutes.
  - 3.1.3 Place in ultrasonic bath for 30 minutes.
- 3.2 Wipes
  - 3.2.1 Add 10 ml hexane to sample container. Add surrogate. Seal.
  - 3.2.2 Place in shaker for 30 minutes.
  - 3.2.3 Place in ultrasonic bath for 30 minutes.
  - 3.2.4 Reduce volume to less than 5.0 ml by blowing down with nitrogen. Bring to 5.0 ml with hexane.
  - 3.2.5 Slowly add 2 ml conc. sulfuric acid, shake, centrifuge.
- 3.3 Oils
  - 3.3.1 Weight out 1.0 g oil, record weight, add 1.0 ml hexane and 1.0 ml surrogate. Vortex.
  - 3.3.2 Place 5.5 g florisil in a 10 ml serological pipet and top with 0.5 g anhydrous sodium sulfate. Elute column with about 50 ml hexane. Discard.
  - 3.3.3 Place 1.0 ml of sample to top of column and elute with 25 ml hexane at 5 ml/min. The PCB's are in this fraction. Transfer to K-D and concentrate to 10.0 ml with hexane. Transfer to 16 ml vial and cap with teflon-lined cap.

4.0 QUALITY CONTROL
- 4.1 Extract one blank per batch or every 20 samples.

4.2 Extract one spike and duplicate per batch or every 20 samples.

EXTRACTION, PESTICIDE / PCB, WATER

1.0 REFERENCE
- 1.1 SW-846, Method 3510
- 1.2 SW-846, Method 3620
- 1.3 EPA 600, Method 608

2.0 REAGENTS
- 2.1 Methylene chloride, pesticide grade, commercial.
- 2.2 Hexane, pesticide grade, commercial.
- 2.3 Acetone, pesticide grade, commercial.
- 2.4 Sodium Hydroxide (10 N) - Dissolve 40g in 100 ml DI water.
- 2.5 Sodium sulfate, reagent grade, commercial.
- 2.6 Sulfuric Acid (1:1) - Slowly add 50 ml acid to 50 ml DI water.

3.0 PROCEDURE
- 3.1 Mark liquid volume in sample container, shake, transfer to 2 L separatory funnel. Adjust pH to 5 - 9. Add surrogate or spike if needed.
  NOTE: TCLP - use 100 ml of TCLP extract, dilute to 1 L with DI water.
- 3.2 Rinse sample container with 60 ml of methylene chloride, transfer to the separatory funnel. Shake for 2 minutes with venting, allow layers to separate for at least 10 minutes.
- 3.3 Measure original sample volume by filling to mark with DI water and measuring volume in graduated cylinder. Record.
- 3.4 Drain the solvent through a funnel containing sodium sulfate into a clean flask.
- 3.5 Repeat extraction two more times and transfer the combined extracts to a K-D apparatus with 3-ball Snyder column. Adjust position in 80-90 C water bath to complete process within 10-20 minutes. Concentrate to less than 1.0 ml.
- 3.6 Remove Snyder and add about 20 ml of hexane, re-attach Snyder and concentrate to approximately 1.0 ml.
- 3.7 Wet Florisil Column with about 1.0 ml hexane/acetone (9:1), transfer extract to top of column containing 3 g of Florisil. Elute with 10 ml hexane/acetone (9:1).
  NOTE: For PCB only use hexane.
- 3.8 Adjust the final volume of eluate to 10.0 ml with hexane, transfer to amber vial.

4.0 QUALITY CONTROL
- 4.1 A blank is extracted with each batch or 20 samples.
- 4.2 A matrix spike and spike duplicate are extracted with each batch or 20 samples.

Written by: Michael D. Quinn Approved by: Theodore J. Ouellette

S.O.P. No. 30
REV. DATE: 11/4/97

FLASH POINT AND IGNITABILITY

1.0 REFERENCE
- 1.1 SW-846, Method 1010
- 1.2 SW-846, Chapter Seven (40 CRF 261.21, 49 CRF 173.3)
- 1.3 ASTM D4982, D93 and G515.5

2.0 REAGENTS
- 2.1 p-xylene (flash pt. 81 =/- 4 F), reagent grade, commercial source.

3.0 PROCEDURE
- 3.1 Ignitability (solids):
  - 3.1.1 Spread about 10 g of sample in the shallow, metal test dish. Wet starch/iodide paper and touch sample. If color changes the sample is an oxidizer and is considered ignitable.
  - 3.1.2 Place sparking igniter adjacent to sample. Attempt ignition 3 times. The sample is considered positive if it ignites.
  - 3.1.3 If sample fails to ignite, touch it with open flame. The sample is ignitable if it burns vigorously. Place about 10 to 20 g in tester being sure temperature probe is under sample surface, disable stirrer, start test and check flash at 80 F and at least every 10° F up to 200° F. Record temperature of flash. Repeat test if flash is detected.

- 3.2 Flash Point (liquids):
  - 3.2.1 Open the gas valve and ignite flame on tester. Note: use of electric igniter is acceptable.
  - 3.2.2 Fill the cup to the fill line. This line is about 2/3 full.
  - 3.2.3 Replace lid onto cup and switch stirrer on. On automated unit load US1 method and press "run" key.
  - 3.2.4 Begin controlled heating.
  - 3.2.5 The flame is introduced to the cup at 10° intervals or less up to 200°F. Record value displayed if flash occurs. If flash occurs repeat test.

4.0 QUALITY CONTROL
- 4.1 The samples that flash are duplicated to confirm flash and temperature.
- 4.2 Each batch check using p-xylene, f.p. 81°F + 4°F. Run in duplicate, record.
- 4.3 Avoid excessive drafts.

Written by: _____ Approved by: _____

4.4 Adjust flame of tester to a near blue color about 1/2 inch in length.
4.5 Correction for varying barometric pressure is not needed when using +/- 10 F.
4.6 To correct for varying barometric pressure use the following:
corrected flash point = observed flash F + 0.06 (760 - barometric pressure)
4.7 If barometric pressure is < 760 mm round up to nearest 1 F, if > 760 round down.
4.8 The automated tester automatically corrects for barometric pressure, do no correct results manually.
4.9 All test must be started with a block temperature less than 80 F and a sample temperature less than 75 F.
4.10 Clean sample cup and temperature probe between each sample by rinsing with acetone, wiping with towel, followed by a water rinse to remove acetone and gently blotting dry with clean cloth.

METHOD 8151 A

HERBICIDES - CHLORINATED BY GC-ECD

1.0 SCOPE

1.1 This method describes the analysis of derivatized chlorinated herbicides by capillary column gas chromatography with ECD. It applies to aqueous, soil and waste matrices. Samples are extracted with diethyl ether and then derivatized with diazomethane.

2.0 INTERFERENCES

2.1 Most interferences are due to coextractable organics like chlorinated acids.

3.0 REAGENTS

3.1 Hexane, Pesticide Grade, commercial source.

3.2 Herbicide Standards (free acid and esters), Ultra (HBM-8150A-1, HBM-8150M-1) or equivalent. Store in teflon-lined, amber vials at 4 C. Free acids are good for two months, derivatives are good for one year.

3.3 2,4-Dichlorophenyl Acetic Acid (free) and Methyl ester , surrogate, Ultra (PPS-165-1, PPS-166-1) or equivalent. Prepare a 10 ug/ml surrogate standard.

3.4 Methanol and Acetone, pesticide grade, commercial source.

4.0 PROCEDURE

4.1 TCLP herbicides, 2,4-D and 2,4,5-TP, are run isothermal on a 1.5% SP-2250/0.95%SP-2401 packed column at 188 C, injector at 250 C and ECD at 310 C, analysis using dual capillary columns, 30 m x 0.53 mm SPB-5 and SPB--608 or equivalent with ECD is acceptable. No surrogate is required for TCLP.

4.2 For priority pollutant herbicides, Method 8150 only, a packed 10% OV-210 column and a temperature programmed run may be used. Use the following temperature program: initially 125 C, hold 3 minutes, ramp at 10 C/min., final 215 C, hold 5 min.

4.3 To analyze priority pollutant herbicides, Method 8150 and/or 8151 by dual capillary load Method 2 into either PE#4 or TP-1 GC and load "8150PE#4.met" or "8150TREM.met" or "Herb Pe#4.met" / "HerbTREM.met" for 8151 into the PC. The following temperature program is used: initial 110 C, hold 3 min., ramp 5 C/min, final 260 C, hold 5 min. Set injector to 225 C and detectors to 350-375 C. Column flow rates are 7.0 ml/min, 75 ml/min make-up of Nitrogen. Primary column - 30 m x 0.53 mm, RTX-5, 0.5 um df, Restex or equivalent. Confirmation column - 30 m x 0.53 mm, SPB-50, 0.5 um df, Supelco or equivalent.

4.4 Prepare calibration standards using herbicide free acid mix , derivatize prior to

METHOD 8151 A

analysis. For spiking use 100 ul of stock free acid standard to 10 ml final extract volume. Prepare intermediate surrogate standard by adding 10.0 ul of 5000 ug/ml stock free acid standard to 10.0 ml acetone for 5.0 ug/ml standard. Use 1.0 ml surrogate for a 0.5 ug/ml concentration in the final extract.

4.5 Prepare calibration standards by diluting the stock herbicide free acid standard, after methylation, as follows:

Cal. Std. 1 = 5 ul to 995 ul hexane (conc. in mix varies, see certification for each component)
Cal. Std. 2 = 10 ul to 990 ul hexane
Cal. Std. 3 = 20 ul to 980 ul hexane
Cal. Std. 4 = 25 ul to 975 ul hexane
Cal. Std. 5 = 50 ul to 950 ul hexane

4.6 Successful calibration requires all RSD's to be less than 20. Verify calibration by analyzing a mid-range methylated standard prepared from a different source. %D must be equal or less than 15.

4.7 During the analysis include a check standard every 10 samples and at the end. The standard must be within 15% or repeat all subsequent analysis after correcting the problem.

5.0 QA/QC

5.1 Every batch or every 20 samples requires a method blank, matrix spike, matrix spike duplicate and a laboratory control sample (LCS). Spike, LCS and surrogate recovery for water should initially be 70-130% and soils 70-130% or use laboratory statistically derived limits. If not check integration, re-analyze, re-extract and re-analyze.

5.2 All target compounds must be below PQL in blank.

5.3 Calculate results as follows:

$$\text{conc. (mg/L or mg/kg)} = \frac{\text{(area or ht. of sample)}}{\text{(area or ht. std.)}} \times \text{conc.std.} \times \text{dilution} \times \frac{\text{(vol. of extract)}}{\text{(vol. or wt. extracted)}}$$

NOTE: integrated result replaces area sample/area std. x conc. std. in above equation

5.4 Sample extracts must be stored at 4 C and protected from light.

5.5 Water samples must be extracted within 7 days, soils within 14 days. Extracts must be analyzed within 40 days.

6.0 REFERENCE

6.1 SW-846, Methods 8150 and 8151A

6.2 Standard Methods, 17th Edition, Method 6640

LACHAT QUIK CHEM AUTOANALYZER PROCEDURE

1.0 GENERAL INSTRUMENT OPERATION

1.1 Start-up

1.1.1 Turn on power strip for the autosampler, pump, and computer.

1.1.2 Install manifold, each is labeled with QuikChem method number and name of the analyte and contains a sample loop, interference filter and appropriate pump tubes. See Table 1.

TABLE 1

Analyte	Wavelength	Channel
Ammonia	630 nm	1
Chloride	480 nm	2
Cyanide	570 nm	1
Nitrate/Nitrite	520 nm	1
Nitrite	520nm	2
Phenols	500 nm	2
Sulfate	420 nm	1
TKN	660 nm	2
Tphos.	880 nm	2

1.1.3 Reagent Water Check - Place all pump tubes in DI water, turn pump on. Inspect manifold for leaks or air bubbles. Correct before beginning run.

1.2 Shut-down - Place all lines in DI water, rinse for 5 minutes then allow all liquids to be pumped out of the manifold. Release the pump tube cartridges tension. Switch off the master power strip.

2.0 Computer Operation

2.1 Log in Omnion and open the method you want to run.

2.2 Create your new tray and save it under a specific name.

2.3 Run the analysis by clicking on the icon "(run tray)". When the analysis is complete this icon will turn from (Stop) back to (run tray).

2.4 View the calibrations and review data.

2.5 Print a report for your results.

3.0 DAILY MAINTENANCE

3.1 Instrument inspection - Inspect and clean surfaces on the autosampler.

3.2 Valves - Check for any blockages or improper working at start of every run. If there is a problem, clean ports and O-rings or replace.

3.3 Pumps - Inspect pump tubing, replace with new pump tubes as required.

ION CHROMATOGRAPHY - ANIONS

1.0 REFERENCE

1.1 SW-846, Method 9056, Rev. 0, 1994

1.2 EPA Method 300 (Drinking Water), Rev. 2.1, 1993

1.3 Standard Methods 17th Ed. Method 4110 B

2.0 REAGENTS

2.1 Dionex AS-14 Column - Eluent stock solution: Dissolve 50.88 g sodium carbonate and 5.04 g sodium bicarbonate in 1 L DI. To run dilute 8.0 ml of eluent stock solution to 1 liter with DI water. Set flow rate to 1.5 ml/min. Range at 100 uS. Use 250 ul sample loop.

2.2 Fluoride, Chloride, Bromide, Nitrate, Sulfate standards, reagent grade, commercial. Stable for one month when stored at 4 C. Diluted working standards good for one week except for nitrite and phosphate which should fresh daily.

2.3 Nitrite (1000 mg/L): Dissolve 4.9257 g of sodium nitrite in 1 L DI water. NOTE:Nitrite easily oxidizes, may require frequent prep. Commercial source acceptable.

2.4 Phosphate (1000 mg/L; PO4-P) - Dissolve 4.3937 g of potassium phosphate (KH_2PO_4) in distilled water and dilute to 1 L. NOTE: This method only gives ortho-Phosphate, not total Phosphate. Commercial source acceptable.

2.5 Regenerant Solution - prepare 500 ml of 0.05N sulfuric acid by adding 0.7 ml of conc. H_2SO_4 to 500 ml volumetric and diluting to mark with DI.

2.6 For perchlorate use a Dionex AS-11 column and 100 mM NaOH eluant. Prepare by diluting 8.0 g of 50% NaOH to 1 L with DI. Use 100 ul sample loop.

3.0 PROCEDURE

3.1 The instrument is turned "ON" by toggling the power switch to the "ON" position and opening valve on air tank. The tank pressure should be about 100 psi. Set range to 100 uS. Flow rate should be 1.5 ml/min (0.9 on pump) for anions except perchlorate. For perchlorate flow rate should be 1.0 ml/min (0.6 on pump).

3.2 Conductivity reading should stabilize in about 5 minutes, adjust to 2.0.

3.3 Verify calibration by injecting the mid-range standard. %D must be less than 10 or correct problem/recalibrate.

3.4 Recalibrate by preparing a four point calibration curve i.e. cal = 1, using specified anion standards. Calculate and record linear regression and correlation coefficient for each anion.

STANDARD CONCENTRATIONS

ANION	STD.1 (mg/L)	STD.2 (mg/L)	STD.3 (mg/L)	STD.4 (mg/L)
F	0.1	1.25	2.5	5.0
Cl	1.0	5.0	10.0	20.0
NO2-N	0.1	1.25	2.5	5.0
Br	1.0	6.25	12.5	25.0
NO3-N	0.1	1.25	2.5	5.0
SO4	5.0	10.0	20.0	40.0

For perchlorate the low standard should be 0.7 ug/ml to 7 ug/ml. Three points are acceptable. Use perchloric acid for primary standard (69 %).
Verify calibration with a mid-level second source standard or different lot. %D must be 10 or less or recalibrate.

3.5 Filter water samples through 0.45 um filter, GPH Acrodisc 13 or equivalent, for soils prepare a 1:10 dilution with warm DI water and mixed for 10 minutes, allow to settle and filter supernate, inject samples using the same technique as for the blank and standards.

3.6 The instrument is shut down by toggling the power switch to the "OFF" position and closing the valve on the air tank.

4.0 CALCULATIONS

4.1 Water - anion concentration is read directly off the integrator. Multiply result by any dilution used.

4.2 Soils/Solids - anion concentration is read directly off integrator. Multiply by 10 and any additional dilution.

5.0 QUALITY CONTROL

5.1 A blank taken through entire prep procedure is run for each sample batch, target anion must be less than PQL.

5.3 One sample in every ten is duplicated and one matrix spiked/matrix spike duplicate for each sample batch not to exceed 20 samples. %RPD less than 20.

5.4 A mid-point verification standard and blank is analyzed at the beginning, after every 10 injections, and at end of sequence. If %D greater than 10 repeat subsequent samples.

5.5 MDL's must be run every 6 months. Analyze seven consecutive replicates of Std. 1, calculate MDL by multiplying standard deviation of replicates by 3.14.

METHOD 7470/7471/245

MERCURY - COLD VAPOR (LEEMAN PS-200)

1.0 SCOPE AND APPLICATION

This procedure is suitable for the determination of mercury by cold-vapor atomic absorption in aqueous wastes, mobility-procedure extracts, ground water and solid-sludge samples.

2.0 SUMMARY OF METHOD

The cold-vapor technique is based on absorption at 253.7 nm. Mercury is reduced to its elemental state and aerated in a closed system. Absorbance is a function of concentration. Typical detection limit is 0.0002 mg/L and 0.1 ug/g.

3.0 APPARATUS AND MATERALS

3.1 Leeman Mercury Analyzer

4.0 INTERFERENCES

4.1 Potassium permanganate and hydroxyamine sulfate minimizes interference from sulfide and chloride.

4.2 Certain volatile organics may cause interference, if suspected, purge and analyze without adding reagents.

5.0 REAGENTS

5.1 Sulfuric acid, conc., ACS grade, commercial source.

5.2 Nitric acid, conc., ACS grade low mercury, commercial source.

5.3 Stannous chloride - Dissolve 100 g of stannous chloride in 1000 ml in 5% HCL. Stir continously during use.

5.4 12% sodium chloride - 12% hydroxylamine sulfate - Dissolve 120 gm of NaCl and 120 gm of hydroxylamine sulfate in 1 liter DI water.

5.5 Potassium Permangate (5 %) - dissolve 50 g in 1000 ml DI water.

5.6 Potassium persulfate (5 %) - Dissolve 50 g of potassium persulfate in 1000 ml of DI water.

5.7 Aqua-Regia - prepare immediately before use, add 3:1 conc. HCL to conc. HNO_3.

5.8 Mercury Stock Standard (1000 mg/L), certified, commercial source.

5.9 Mercury Working Standard (1.0 mg/L) - Dilute 0.05 ml stock to 50.0 ml with 5 % HN03.

METHOD 7470/7471/245

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Mercury free plastic or glass containers are suitable.

6.2 Aqueous samples must be acidified to pH < 2 using HNO3. Maximum holding time is 28 days.

6.3 Non-aqueous samples shall be refrigerated and analyzed as soon as possible.

7.0 PROCEDURE

7.1 Digestion Procedure

7.1.1 Waters - Prepare calibration standards of 5.0 (0.25 ml), 2.0 (0.1 ml), 1.0 (0.05 ml), 0.5 (0.025 ml), 0.2 (0.01 ml) and 0.0 ug/l mercury in 50 ml of 5% HN03. Transfer 30 ml of standards, samples and QC samples in 50 ml polypropylene centrifuge tubes. Add 0.75 ml conc. nitric acid, 1.5 ml conc. sulfuric acid, mix, add 4.5 ml of 5% $KMNO_4$, mix, let stand for 15 minutes. Add additional permanganate until purple color persists. Add 2.4 ml of potassium persulfate and heat for 2 hour in a water bath maintained at 95° C. Cool and add 1.8 ml of sodium chloride-hydroxylamine sulfate solution. Additional hydroxyamine solution may be needed to complete decolorization. The sample is ready to be analyzed. All samples and standards must contain same volume of reagents. Analyze all samples and standards in duplicate. Report average.

7.1.2 245.5 CLP-M (SOIL) - Prepare calibration standards of 5.0 (0.5 ml), 2.0 (0.2 ml), 1.0 (0.1 ml), 0.5 (0.05 ml), 0.2 (0.02) and 0.0 ug/l of mercury. Weigh a representative 0.2 g portions of wet sample and place in the bottom of a 50 ml polypropylene centrifuge tube. Add 10 ml DI water, add 5 ml of conc. sulfuric acid and 2.5 ml of conc. nitric acid mixing after each addition. Heat for 2 minutes in a water bath at 95° C. Cool, add 50 ml DI water, add 15 ml potassium permanganate and 8 ml of potassium persulfate. Mix and place in the water bath for 30 minutes at 95 C. Cool, add 6 ml or sufficient amount of sodium chloride-hydroxylamine sulfate to decolorize. Pour reduced sample in 100 ml volumetric flask and fill to mark with DI water. Samples are ready for analysis. Analyze all samples and standards in duplicate, report average. Report as dry weight.

7.1.3 SW-846 Method 7471 (SOIL) - Prepare calibration standards of 5.0 2.0, 1.0, 0.5, 0.2 and 0.0 ug/l of mercury. Weigh three randomly selected 0.2 g portions of sample and place each in the bottom of a 50 ml polypropylene centrifuge tube ie. 0.6 g total. Use 0.2 g for feeds. Add 5 ml of DI water and 5 ml of aqua regia. Heat 2 minutes in a water bath at 95° C. Cool, then add 10 ml DI water and 15 ml of potassium permanganate solution to

METHOD 7470/7471/245

each sample. Mix and place in water bath for 30 minutes at 95° C. Cool, add 6 ml or sufficient amount of sodum chloride-hydroxylamine sulfate to decolorize, transfer reduced sample to a 100 ml volumetric flask fill to mark with DI water. Samples are ready for analysis. Analyze all samples and standards in duplicate, report average.

7.2 GENERAL INSTRUMENT OPERATION

7.2.1 Computer Start-up - Press the MENU key and type "T" then "4", the instrument will turn on and go through a warm-up period. It will flash "instrument ready" on the screen.

7.2.2 Calibration - Put the appropriate standards for the method selected in the standard rack. Press the MENU key and type STANDARD 1-6, 1 REPLICATE, the instrument will begin calibration. After the standards have been run, go back to main menu and select calibration. The new curve be displayed, if its correlation coefficient >0 .995 then type "A" and the new curve will be stored.

7.2.3 Running Samples - From the main menu, select "AUTOSAMPLER" and then "RACK" entry. Enter rack name, the sample ID into the appropriate cup position. Return to the main menu, select AUTOSAMPLER and then SET- UP. Type the rack number to be un (1 or 2), then type the begin cup number and the end cup number. To begin the run press F8, the instrument will begin the sequence.

7.2.4 Shut-down Procedures - Press the MENU key, type "T" then "6", press enter. Turn off the power to the lamp if the instrument will not be used for 24 hours or longer.

8.0 QUALITY CONTROL

8.1 Calibration should consist of six points with a correlation coefficient > 0.995. A calibration curve must be made for every hour of continuous operation.

8.2 Calibration Verification - A verification standard made from different source must be run immediately after calibration. Standard should fall within 10% of true value or recalibrate.

8.3 Continuing Calibration - Every ten samples a blank and mid-level standard should be analyzed and fall within 10% of true value. If not, correct problem, recalibrate and rerun all affected samples.

8.4 A blank and LCS should be analyzed every batch of no more than 20 samples. A spike and spike duplicate should be analyzed every batch or every ten samples. Spike recovery is 85-115 % with an RPD of <20. If recovery is outside limits then the method of standard additions must be used.

8.5 Calculate soil concentration as follows:

METHOD 7470/7471/245

$$ug/g = \frac{\text{(avg. ug/L from cal. curve)(0.1 L)(dilution)}}{\text{wet or dry wt. in g}}$$

8.6 Water concentration , mg/L = (avg. result from cal. curve,ug/L)(dilution) / 1000

8.7 Dilution test, for every batch select one sample containing mercury at least 25 times the detection limit and make a 1: 5 dilution and reanalyze. Agreement should be within 10 %. If not, use standard additions. If all samples are below 10 times the detection limit then use perform spike recovery. Recovery should be 85 - 115 % or use standard additions.

8.8 For standard additions use the single point method. Take two identical aliquots (Vx), to the first labeled A add a known volume (Vs) of known concentration (Cs). To the second aliquot labeled B add the same volume (Vs) of solvent. Analyze both A and B. Calculate unknown concentration (Cx) as follows:

Cx = (Signal B)(Vs)(Cs) / (Signal A - Signal B)(Vx)

9.0 REFERENCE

9.1 Method 245.2 and Method 245.2 CLP-M (automated cold vapor)
9.2 Method 245.5 and Method 245.5 CLP-M (automated cold vapor)
9.3 SW-846 Method 7470
9.4 SW-846 Method 7471

METHOD 6010/200.7

METALS - ICAP

1.0 SCOPE AND APPLICATION

This method uses ICP-AES to measure metals in liquids and solids. It is suitable for the following metals: aluminum, antimony, arsenic, barium, beryllium, boron, cadmium, calcium, chromium, cobalt, copper, iron, lead, lithium, magnesium, manganese, molybdenum, nickel, phosphorus, potassium, selenium, silica as silicon, silver, sodium, strontium, thallium, tin, vanadium and zinc. All matices including water, TCLP extracts, organic wastes, soils, sludges, sediments and other wastes require digestion prior to analysis.

2.0 SUMMARY OF METHOD

Prior to analysis samples are acid digested/solubilized. The sample is drawn into a spray chamber, nebulized and ionized in a plasma. Element specific characteristic emission spectra is measured by optical spectrometry. Line intensity is related to concentration. Background correction is used for low-level detection.

3.0 INTERFERENCES

3.1 Background and stray light are compensated for by background subtraction adjacent to peak. Exact location determined by complexity of spectrum adjacent to peak of interest.

3.2 Spectral overlap may be corrected for by using an alternate wavelength or by use of interelement correction factors. The correction factors must be evaluated for each instrument since the intensities will vary.

3.3 Chemical interferences are not normally significant. Highly dependent on matrix.

3.4 Memory effects occur when analytes from previous sample contribute to signal. Suitable rinse time should be used. At least 60 seconds is required.

4.0 APPARATUS AND MATERIALS

4.1 ICP-AES Jarrell-Ash Trace with data aquisition software.

4.2 ICP-AES Jarrell-Ash 9000 with data aquisition software.

4.3 Argon gas, high purity, commercial source.

5.0 REAGENTS

5.1 Reagent water, Type II, in-house DI or commercial, targets less than MDL.

5.2 Nitric and hydrochloric acid (conc.), high purity, commercial source.

5.3 Dilution Acid (waters) - dilute stock standards using 2 ml of (1:1) nitric acid and 10 ml of (1:1) hydrochloric acid per 100 ml DI water.
Soils - dilute 5 ml conc. nitric acid to 100 ml with DI water for a 5 % solution.

5.4 Calibration blank (ICAL-B) - Prepare by acidifying reagent water to the same concentration of acids found in the standards and samples. For ICAL-B, add 5.0 ml of trace metal grade Nitric acid per 100 ml of water.

5.5 ICAL-1 - Dilute 200 ul of SPEX stock ICAL-1 solution to 200 ml 5% Nitric acid solution. This solution contains 5 ppm of Sodium, Calcium, Magnesium and Potassium.

5.6 ICAL-2 - Dilute 2.0 ml of SPEX ICAL-2 stock standard to 200 ml of 5% Nitric acid solution. This solution contains 4 ppm Nickel, 2 ppm Zinc, 1.5 ppm Manganese, 1.0 ppm Silver, and 1.0 ppm Chromium.
NOTE: standard must be stored in dark bottle or in dark location.

5.7 ICAL-3 - Dilute 2.0 ml of SPEX ICAL-3 stock standard to 200 ml of 5% Nitric acid. This solution contains 5 ppm Cobalt, 5 ppm Vanadium, 2.5 ppm Copper, and 0.5 ppm Beryllium.

5.8 ICAL-4 - Dilute 4.0 ml of SPEX ICAL-4 stock standard to 200 ml of 5% Nitric acid solution. This solution contains Arsenic and Thallium at 2.0 ppm and Cadmium, Lead and Selenium at 1.0 ppm.

5.9 ICAL-5 - Dilute 500 ml of SPEX ICAL-5 stock standard to 200 ml of 5% Nitric acid solution. This solution contains Antimony at 1.5 ppm.

5.10 ICAL-6 - Dilute 1.0 ml of SPEX ICAL-3 stock standard to 200 ml of 5% Nitric acid solution. This solution contains 5 ppm Iron, 10 ppm Barium and Aluminum.

5.11 ICAL-7 - Dilute 0.2 ml of individual 1000 ug/ml stock standards of Strontium, Tin, Titanium, Molybdenum, Lithium, Bismuth and Boron with 5 % nitric acid to 200 ml for a 1.0 ug/ml each standard.

5.12 INTERFERENCE CHECK SOLUTIONS (ICSA, ICSAB) - Dilute 20 ml of SPEX INT - A1, which contains the interferents and 2 ml of INT-B1 and INT-B2, which contains the analytes to 200 ml with 5% Nitric acid. Dilute 20 ml of SPEX-INT-A1 to 200 ml with 5% Nitric Acid.

5.13 CONTRACT REQUIRED DETECTION LIMIT STANDARD (CRDL) - Dilute 200 ul of SPEX contract required detection limit stock standard to 200 ml with appropriate dilution acid. This solution contains the elements at the required detection limit.

5.14 All working standards are acceptable for use for one month. Any standards containing Silver must be stored in dark bottles or in a dark location.

5.15 Internal standard solution, 5.0 ug/ml Yitrium – dilute 5.0 ml of 1000 ug/ml stock to 1 L with DI water containing 50 ml conc. nitric acid.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 All aqueous samples should be collected in plastic and acidified with HN03 to a pH < 2.0. Analyze within 6 months.

6.2 Solid samples should be collected in 4 oz. glass jars. Analyze within 6 months.

7.0 PROCEDURE

7.1 ICP-AES Model 61-E

7.1.1 Computer start-up - Turn on the power to the printer, CRT and computer. The software will load and a menu will appear. Choose required item number.

7.1.2 Spectrometer start-up

7.1.2.1 Flip the argon toggle switches for the torch and sample argon supplies.

7.1.2.2 Aspirate DI into the nebulizer. Turn on the peristaltic pump.

7.1.2.3 Confirm that the automatic power control is in the manual position and the forward power knob is rotated fully counter-clockwise. Press the button for "RF Generator" power.

7.1.3 Plasma Ignition

7.1.3.1 After the argon has been flowing for five minutes, turn off the pump and sample argon flow using their toggle switches.

7.1.3.2 Increase the forward power to 0.5 KW and press the ignitor button.

7.1.3.3 While holding down the ignitor button, slowly increase the power until the plasma ignites with an audible pop. Release the ignitor.

7.1.3.4 Turn on the sample argon flow and re-start the sample pump.

7.1.3.5 Turn the power control to "ON."

7.1.3.6 Turn the forward power control fully clockwise. It should read 1.1 KW.

7.1.4 Shut Down

7.1.4.1 Press the "RF OFF" button to extinguish the plasma.

7.1.4.2 Allow distilled water to pump for 5-10 minutes.

7.1.4.3 Turn off the gas toggles and the pump.

7.2 ICP-AES Model TRACE

7.2.1 Computer start-up - Turn on the power to the printer, CRT and computer. The software will load automatically and an operating menu will appear.

7.2.2 Spectrometer start-up and plasma ignition.

7.2.2.1 Connect peristaltic pump tubing.

7.2.2.2 Turn on ventilation fan.

7.2.2.3 Select "set-up" from the operations menu and select "plasma

control panel". Press the F1 key, then press the F9 key to engage the plasma.

7.2.2.4 Once the plasma has been initiated, check the pump rate to ensure proper sample flow into the spray chamber.

7.2.2.5 Select a daily operating method to set plasma parameters and begin the peristaltic pump.

7.2.2.6 Profile the instrument by pressing "PROFILE" from main menu, analyze a 5 ppm arsenic standard, instrument will find centroid, measure intensity, adjust if difference exceeds 5 %.

7.2.3 Shut-Down

7.2.3.1 Return to "plasma control panel" and press F7 key to automatically shutdown the plasma and spectrometer systems. Wait for 30 seconds to dissipate remaining heat and turn ventilation fan off. Release pump tubing.

7.3 ANALYSIS

7.3.1 Instrument Warm-up - The computer, spectrometer, RF generator and plasma should be on for at least 30 minutes before analysis begins.

7.3.2 Computer Operation

7.3.2.1 Accessing the analysis program file -
The menu of options will appear at the top of the screen. When the system comes on-line, "operation" is chosen by simply pressing the enter key. By pressing the enter key again, the "analysis" mode is accessed. The software will ask for the method. The method for most analyses will be "routine". Enter "routine" and previously programmed set of element channels, associated parameters will be brought from the disc to memory.

7.3.2.2 Standardization/Calibration -
The insert menu at the bottom of the screen allows you to press the F3 function key to enter the standardization mode. Enter internal standard. A list of required standards will appear for the current method in use. Aspirate an appropriate standard and press F1. After the instrument acquires the data for that standard, press the F9 key to accept the data. Move to the next standard and repeat the process. When all of the standards have been analyzed, press the F9 key to accept all data. The computer will calculate the slope for all channels and display those results.

7.3.2.3 Sample Analysis -
Aspirate the sample to be analyzed and press the F1 key. Enter all of the necessary information pertaining to the sample and press the F1 key. The instrument will acquire the necessary data and will print the results, in concentration on the line printer. Rinse the system with calibration blank for at least 1minute and repeat process for all samples.
Note: to determine dissolved metals filter through 0.45 um pore membrane filter at time of collection or before acid preservation.

7.3.2.4 Dilute and reanalyze samples that exceed 90 % the upper linear range.

8.0 QUALITY CONTROL

8.1 Method 6010

8.1.1 The calibration curve must consist of at least a blank and one standard. Immediately after daily calibration analyze a second source check standard (ICV) and a calibration blank. After every tenth sample and at the end of the sample batch analyze an ICV or CCV. All results must be within 10 % of true with a relative standard deviation of <5 % of at least two integrations or recalibrate. Between analysis of standards and samples flush the system at least one minute using the calibration blank solution.

8.1.2 After initial calibration the highest standard should be reanalyzed and the result must be within 5% of the actual value of the standard or recalibrate.

8.1.3 The results of the calibration blank must be within three standard deviations of the mean blank value. If not, the instrument should be recalibrated and all samples rerun from last verification standard that was within range.

8.1.4 Analyze the interference check solutions at the beginning and end of every shift or twice every 8 hours, whichever is more frequent.. The analyzed value should be within 20% of their true values. If these fall outside range, it may be necessary to determine and adjust the interelement correction factors.

8.1.5 One method blank, pre-digested matrix spike and matrix spike duplicate is required every batch of 20 samples. The batch blank should be below client required detection limit. The duplicate values should be within 20% of the original sample, the spike recoveries should fall within 75 - 125 %.

8.1.6 A sample with a concentration at the CRDL or reporting limit for each analyte should be analyzed at the beginning and end of every daily run. Results must be within 15 % true or recalibrate and reanalyze all effected samples.

8.1.7 One sample of each batch should be diluted 1:4 and re-analyzed if the analyte concentration is at least ten times the instrument detection limit. The value corrected for dilution should read within 10% of the value determined in the undiluted sample.

8.1.8 One post-digestion spike per batch should be analyzed, recovery should be 75-125% of known value. The spike level should be at least ten times but less than 100 times the instrument detection limit.

8.1.9 All blanks, standards and samples must be at the same acid concentration.

8.1.10 Store any standards containing silver in a dark bottle or dark location.

8.2 Method 200.7

8.2.1 The linear dynamic range (LDR) must be established for each wavelength used. Determine by analyzing increasing concentrations until the observed concentration is no more than 10% below the stated concentration. Record and file data. Sample concentrations greater than 90% of the LDR must be diluted and reanalyzed. Perform anually.

8.2.1 Calibrate using a blank and one high standard. Verify initial calibration with a second source standard, quality control sample at concentration greater than 1.0 mg/L except Silver which should be less than 0.5 mg/L, the determined mean of replicate analysis must be within 5 % of stated concentration. The blank must be less than the IDL. Flush with the calibration blank between all standards and each sample (min. 60 seconds). Before each batch check linear range by analyzing the highest standard, results must within 5 % of true.

8.2.2 Analyze instrument performance check (IPC) standard and calibration blank every 10 samples and at end of run. Results must be within 5 % immediately after calibration and 10 % thereafter, otherwise reanalyze samples. The blank value must be <IDL. If not, repeat, if out of range stop analysis, correct, reanalyze effected samples.

8.2.3 Verify interelement and background correction factors at beginning, end and at periodic intervals throughout the run, results must be within 1.5 times the std. deviation of the calibration blank mean.

8.2.4 For dissolved analytes filter through a 0.45 um filter.

8.2.5 Spike a sample at 10 % frequency, recovery should be 70-130 %.

8.2.6 Every batch must have a Lab Fortified Blank, spiked with all targets, % recovery must be 85 - 115.

8.2.7 One sample of each batch should be diluted 1:4 and re-analyzed if the analyte concentration is at least ten times the instrument detection limit. The value corrected for dilution should read within 10% of the value determined in the undiluted sample.

8.2.8 One post-digestion spike per batch should be analyzed, recovery should be 85-115% of known value. The spike level should be at least twenty times but less than 100 times the method detection limit.

8.2.9 All blanks, standards and samples must be at the same acid concentration.

8.2.10 Store any standards containing silver in a dark bottle or dark location.

8.3 Calculations:

$$\text{conc. (ug/ml or ug/g)} = \frac{\text{(Inst. readout)(digest vol, ml)(dilution fact.)}}{\text{sample vol, ml or wt. sample, g}}$$

8.4 Torch inspection and cleaning - Daily inspect the plasma torch for salt build-up or other contamination, clean by removing the torch and rinsing with hot, soapy water. Rinse with DI thoroughly before re- installation.

8.5 Nebulizer cleaning - Daily examine to see if any blockage exists. Clean by gently forcing DI water through the tip being careful not to break the capillary.

8.6 Filter Check - The air intake filters, rear, of the instrument should be checked for accumulation of lint or debris. If needed, clean. The filters on the power supply and right side of instrument should be examined and replaced when necessary.

8.7 Pump oil check - Check for proper level. Add oil if needed. Change pump oil yearly, or when the vacuum gauge needle exceeds 15.

9.0 REFERENCES

9.1 SW-846 Method 6010 B, Rev. 2, Jan. 1995

9.2 EPA 600 Method 200.7, Rev. 4.4, May 1994

ORGANOPHOSPHORUS PESTICIDES

1.0 REFERENCE:
- 1.1 SW-846, Method 8140
- 1.2 SW-846, Method 8141

2.0 REAGENTS
- 2.1 Hexane, Pesticide Grade, commercial source.
- 2.2 Organophosphorus Standards, Ultra, Accustandard, Chem Service or other source.
- 2.3 2-Nitro-m-xylene (surrogate), ACS reagent grade, commercial source.

3.0 PROCEDURE
- 3.1 The analysis is performed on 30 m x 0.53 mm RTX-5 or equivalent using the NPD. To operate the detector turn on both the air and hydrogen, press DETECTOR then YES, press NEXT to step through the menu, set source voltage to 5 and press ENTER, press PREV and then ENTER to turn detector ON. Press NEXT and increase the source voltage in increments of 10 to about 50 until a large increase in voltage is observed on the data acquisition system then add 5, to operate at optimum voltage. If detector does not respond contact supervisor. Set the GC injector to 220 C and the detector to 320 C, use 5.0 ml/min helium flow and the following temperature program:
  initial 150 C, hold 10 min, ramp at 4 C/min, final 320 C, hold 5 minutes.
- 3.2 Load OPPEST method into the PC. Load Method 6 (OPPEST) in Tracor TP-1 GC.
- 3.3 Prepare calibration standards by diluting 10 ug/ml intermediate standards. Two standards are used: the OP long (ULTRA # SPM-824) containing 20 components and the OP short (ULTRA # SPM-834) containing seven components. Prepare a 10 ug/ml Malathion spike standard, spike using 1.0 ml to each sample for 1.0 ug/ml in the 10.0 ml extract. Prepare a 10 ug/ml surrogate standard, add 1.0 ml to each sample for 1.0 ug/ml in the 10.0 ml extract. Calibration standards should represent the linear range but the lowest standard must be at 0.1 ug/ml. Problems may occur with TEPP, Dichlorvos, Naled, Trichlorfon, and Merphos. Injection port maintenance is usually needed if breakdown/low response is detected. Both Demeton and Merphos will show two peaks each. Quantitate Demeton using first peak, quantitate Merphos using the sum of it two peaks. Tokuthion should show no breakdown, if detected replace liner and clip guard column.
- 3.4 Manually inject 1 ul of each standard and sample in Injection Port A of TP-1. Press START on GC.

3.5 Due to the specificity of the NPD, limited confirmation of positives is required. If the target compound responds on the ECD, confirm using dual-column pesticide instrument. If the concentration is high, confirm by GC-MS.

3.6 Quantitation should be performed using a single-point standard within 10 % of the target compound. Either electronic or manual quantitation is acceptable. Calculate as follows:

$$\text{conc. (mg/L or mg/kg)} = \frac{\text{(area or ht. sample)}}{\text{(area or ht. std.)}} \times \text{conc. std.} \times \text{dilution} \frac{\text{(vol. of extract)}}{\text{(vol. or wt. extracted)}}$$

4.0 QA/QC

4.1 Surrogate and spike recovery for aqueous sample should be 70-130% and for soils 50-150%.

4.2 Each batch or every 20 samples should have a blank, spike and duplicate.

4.3 Due to the changing response characteristic of NPD's, the requirement for an active five-point calibration is relaxed. Best results are obtained by closely matching standards with the concentration of the target ie. within 10 %. The method of standard additions is also acceptable.

METHOD 8081/8082/608

PESTICIDE/PCB BY GC-ECD

1.0 SCOPE AND APPLICATION

This procedure is used to determine various organochlorine pesticides and PCB's as Aroclors in hexane extracts of solid and liquid matrices. This method may also be used for selected organophosphorus and triazine pesticides.

2.0 SUMMARY OF METHOD

This method uses dual-column capillary chromatography coupled with electron-capture detection to resolve, identify and quantitate single and multi-component target compounds. Samples are extracted with methylene chloride and concentrated, then solvent exchanged for analysis. Various clean-up procedures may be used i.e. sulfuric acid for PCB only samples.

3.0 INTERFERENCES

3.1 Phthalates may be introduced during sampling and extraction, remove by silica gel or Florisil cleanup.

3.2 Sulfur may cause problems with early eluting compounds, remove by TBA method 3660. Note: must determine endrin aldehyde prior to this cleanup.

4.0 APPARATUS AND MATERIALS

4.1 Prekin-Elmer, Shimadzu and Tremetrics GC each with dual ECD's.

4.2 EZChrom Data Aquisition System

4.3 Dual capillary system - uncoated 5 m gaurd column, y-pressfit, 30 m x 0.53 mm RTX-5 and 30 m x 0.53 mm RTX-50 or equivalent. Option: 0.32 mm acceptable.

5.0 REAGENTS

5.1 Pesticide/PCB stock standards: Ultra, Supelco, Accustandard or equivalent. Store stock standards at 4 C in PTFE sealed containers in the dark. Good for 1 year. All others good for 6 months.

5.2 Surrogates: TCMX and decachlorobiphenyl, commercial source.

5.3 Methylene chloride, Hexane and Acetone: Pesticide Grade, commercial source.

5.4 Tetrabutylammonium Hydrogen Sulfate, commercial source.

5.5 Sodium Sulfite, commercial source.

5.6 2-Propanol; Reagent Grade, commercial source.

5.7 Sulfuric Acid, Reagent Grade, commercial source.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Collect aqeuous samples in 1 L amber glass with teflon-lined cap. Refrigerate. Extract within 7 days, analyze extracts within 40 days. Store refrigerated in the dark.

6.2 Collect solid/soil samples in 4 or 8 oz. glass jar with teflon-lined cap. Refrigerate. Extract within 14 days, analyze extract within 40 days. Store refrigerated in the dark.

7.0 PROCEDURE

7.1 Pesticides

7.1.1 Set GC to injector to 250 C and ECDs to 340-375 C. Set capillary flow rates to about 5 ml/min. with 30 ml/min make-up of Nitrogen. The following temperature program is used: initial 150 C, hold 5.0 min, ramp at 7 C/min, final 290 C, hold 10 minutes.

7.1.2 Five-point calibration curves are required with a RSD of less than 10% for EPA 608 or 20% for 8081 for each target compound. Calibration should represent the linear range. The following range is recommended: 0.005, 0.01, 0.020, 0.05, 0.08 ug/ml for individual pesticides and 0.1. 0.25, 0.5, 1.0 and 2.0 ug/ml for Toxophene and Chlordane (tech). All calibrations should include applicable surrogates. Verify initial calibration using a mid-level standard prepared from a second source. %D=/<15 or recalibrate.

7.1.3 Measure, before samples are analyzed and every 12 hours, percent breakdown by checking DDT and Endrin (0.05 ug/ml each) standard and a mid-level check pesticide check standard, each must be less than 15%. If either fails, clean liner, clip guard column, etc. Repeat and/or re-calibrate.

7.1.4 Prepare working standards from stock standards as follows: Dilute 50 ul of 2000 ug/ml pesticide mix (Supelco #4-8913), add 200 ul of 500 ug/ml TCMX, 200 ul of 500 ug/ml DCB 9.60 ml hexane for a 10 ug/ml each standard. Take 1.0 ml of this intermediate standard and dilute to 10.0 ml with hexane for a 1.0 ug/ml working standard. Prepare daily standards and calibration standards by serial dilution: 80 ul to 1.0 ml hexane for 0.08 ug/ml; 50 ul in 1 ml for 0.05 ug/ml; 20 ul in 1 ml for 0.02 ug/ml; 10 ul in 1 ml for 0.01 ug/ml; and 5 ul in 1 ml for 0.005 ug/ml. Prepare a 5 ug/ml surrogate and pesticide spike standard, 1.0 ml in the 10.0 ml extract results in a 0.05 ug/ml concentration each. At a minimum report spiked recoveries for Lindane, Heptachlor, Aldrin, Dieldrin Endrin and DDT.

7.1.5 Highly-colored extracts may require dilution. It is recommended that soil extracts be sulfur cleaned as follows:

7.1.5.1 Prepare tetrabutylammonium sulfite reagent by mixing 1.36 g of tetrabutyl ammonium hydrogen sulfate and 10 g sodium sulfite in 40 ml DI water. Shake well. Store in amber vial for up to one month.

7.1.5.2 To 1.0 ml of sample extract add 1.0 ml of sulfur reagent (3.6.1) and 1.0 ml of 2-propanol, cap and shake. Solid crystals should be visible, if not add 0.1 g of sodium sulfite until crystals remain after shaking. Ad 5.0 ml of DI water and shake, allow to separate.

7.1.5.3 Transfer top hexane layer to micro-snyder or using nitrogen blowdown concentrate to 1.0 ml. This extract is ready for analysis.

NOTE: This clean-up degrades Endrin Aldehyde.

7.1.6 For pesticides in oils/organic wastes take 1.0 g of sample and dilute to 20.0 ml with hexane, add 2.0 ul of 500 ug/ml surrogates for a 0.05 ug/ml concentration. Shake and allow phases to separate. Perform sulfur clean-up and/or florisil cleanup. Place in appropriate autosampler and start run after data system is initiated. Calculate surrogate recovery.

7.1.7 For pesticide analysis by capillary GC on PE#4 load GC Method 1, on TP-1 load GC Method 1 (PEST) and load method 1 on Shimadzu. In PC load "PESTPE4" or "PESTTREM" or "PESTSHIM", set up run sequence and load sequence batch run. Check for "Waiting for Trigger" message.

7.1.8 Place sample and standards in autosampler. Check that the correct PC method has been loaded and that the batch sequence has been entered. To start the PE GC press AUTO then move the arrow to START and press ENTER. For the Tracor GC press START on the HP autosampler. For the Shimadzu press START on the autosampler.

7.1.9 For TCLP make a 20 x dilution of extract, i.e. 50 ul to 950 ul hexane.

7.1.10 Electronic calculations are recommended however, if manual integration is needed use the following equation:

$$\text{conc. (ug/ml or mg/kg)} = \frac{\text{(area sample)}}{\text{CF}} \times \text{conc. std.} \times \text{dilution} \times \frac{\text{(extract vol. in ml)}}{\text{(vol. or wt. extracted)}}$$

$$\%\ \text{BREAKDOWN DDT} = \frac{\text{(area of DDE + DDD)}}{\text{(area of DDT + DDE + DDD)}} \times 100$$

$$\%\ \text{BREAKDOWN ENDRIN} = \frac{\text{(area of endrin ald. + endrin ketone)}}{\text{(area of endrin + ald. + endrin ketone)}} \times 100$$

$$\% \text{ RECOVERY} = \frac{\text{(conc. found)}}{\text{(spike conc.)}} \times 100$$

7.1.11 All multi component compounds, Chlordane (Tech) and Toxaphene, are calculated by summing of several common peaks. For Toxaphene, use at least five major peaks. It is vital that all baselines be equivalent and that identical components are integrated. Due to differences in Chlordane, report Heptachlor/Heptachlor Epoxide, alpha and gamma Chlordane separately if concentrations differs from standard.

7.1.12 Calculate results from both columns and report the higher of the two values. If the results differ by more than 40 % add comment to worklist.

7.2 PCB's

7.2.1 The same column pairs and run conditions are used as for pesticides. The DDT and Endrin breakdown criteria is not applicable. Calibrate using five standards containing a mixture of 1016 and 1260 to show linearity and to use to quantitate 1016 and 1260. Prepare a 10 ug/ml intermediate standard of each Aroclor from the stock standards. Calibrate using 0.1 ug/ml, 0.2 ug/ml, 0.5 ug/ml, 0.7 ug/ml and 1.0 ug/ml. Include surrogates in calibration. Prepare a 0.5 ug.ml standard for the other Aroclors. These are used to aid the analyst in pattern recognition and a single-point calibration factor for quantitation. It is permissible to calibrate with a five-point each Aroclor.

7.2.2 For quantitation at least 3 congeners must be chosen that are characteristic of that specific Aroclor. They must be at least 25 % the height of the largest Aroclor peak. Of the three peaks at least one must be unique to the specific Aroclor. Calculate calibration factor (CF) as follows:

$$CF = \frac{\text{Peak area in standard}}{\text{Total mass of standard}}$$

Five sets of CF's will be generated for the 1016/1260 mixture, each set consisting of at least 5 peaks. The single standards will generate at least three CF's, one for each selected peak.

7.2.3 Tentative identification occurs when the RT of a sample peak falls within the RT window on both columns. Once identified compare the responses of 3 to five major peaks in the single-point standard to the sample. The amount of Aroclor is calculated using the individual calibration factor for each peak chosen and averaging the result. It is acceptable to quantitate using the total area of the identified Aroclor provided exactly the sample peaks are used in

calibration. Any peaks not identified based on retention times must be subtracted from the total area. Calculate concentration as in p. 7.1.10.

7.2.4 Calculate results from both columns, report the highest value. If the results differ by more than 40 % add comment to worklist.

8.0 QA/QC

8.1 All blanks must be less than PQL i.e. 0.005 ug/ml for individual pesticides.

8.2 A mid-level verification standard must be analyzed every 10 samples and at the end or every 12 hours, if %D is greater than 15% all affected samples must be repeated after problem is resolved.

8.3 If surrogate recoveries are not within limits, re-check calculations using area and/or height, examine for interfering peaks or baseline disruptions, re-analyze, and/or re-extract and re-analyze. If recoveries are still out of limits, flag the data.

8.4 A blank, spike and spike duplicate and LCS must be analyzed every 20 samples or batch if smaller. LCS recoveries should be 70-130%. Spike recoveries should be within statistically derived limits with an RPD =/< 20%.

8.5 Retention time windows are used to identify peaks. Establish windows by multiplying by 3 the standard deviation of the RT over a 72 hour period. This must be done on both GC columns and any repeated when new columns are installed or other major changes occur. Use calibration standards analyzed during sequence to evaluate RT stability, if any falls outside window correct problem and repeat effected samples.

8.6 Sample injections may continue for as long as calibration verification meet QC requirements. To quantitate the peak must be at least 2.5 times baseline noise.

9.0 REFERENCES

9.1 SW-846, Method 8082 , Rev.0, Dec. 1996
9.2 SW-846, Method 8181A, Rev. 0, Dec. 1996
9.3 EPA Method 608
9.4 SW-846, Method 3660

PHENOLICS, TOTAL

1.0 REFERENCE

1.1 SW-846, Method 9065A
1.2 EPA 600, Method 420.1
1.3 EPA 600, Method 420.2

2.0 REAGENTS

2.1 Phosphoric acid solution: Dilute 10 ml of 85% phosphoric acid, reagent grade, to 100 ml with DI water.
2.2 Copper sulfate solution: Dissolve 10 g copper sulfate in 100 ml DI water.
2.3 Buffer Solution: Dissolve 16.9 g Ammonium Chloride in 143 ml concentrated Ammonium Hydroxide and dilute to 250 ml with DI water.
2.4 4-Aminoantipyrine solution (manual): Dissolve 2 g 4AAP in 100 ml DI water.
2.5 Potassium Ferricyanide Solution: Dissolve 8 g $K_3Fe(CN)_6$ in 100 ml DI water.
2.6 Stock Phenol Solution: Dissolve 1.0 g phenol in freshly boiled and cooled water and bring volume to 1000 ml with water. (1 ml = 1000 ug phenol).
2.7 Working Phenol Solution: Dilute 0.10 ml of Stock Phenol Solution to 10 ml with DI water. (1 ml = 10 ug Phenol)
2.8 Chloroform: ACS grade, commercial.
2.9 Ferrous Ammonium Sulfate: Dissolve 0.11 g in 50 ml D Iwater containing 0.1 ml concentrated sulfuric acid and dilute to 100 ml with boiled DI water.
2.10 Sulfuric Acid, concentrated reagent grade, commercial.
2.11 4 - Aminoantipyrine color reagent: Dissolve 0.16 g 4 -aminoantipyrine in 250 ml of water.
2.12 Buffered Potassium Ferricyanide, pH 10.3: Dissolve 2 g $K_3Fe(Cn)_6$, 3.I g boric acid and 3.75 g potassium chloride in 800 ml DI water. Add 47 ml of I M NaOH and dilute to I liter with DI water.

3.0 PROCEDURE

3.I Distillation:

3.1.1 Measure 50 ml of sample into a boiling flask. Adjust the pH to < 4.0 with 1:1 H_3 PO4 and add 1 ml $CuSO_4$ solution. For soil, use 5.0 g plus 50 ml of DI water, adjust pH to 4. Distill at 190 C.
3.1.2 Add a couple boiling chips and distill 45 ml of sample. Add 5.0 ml DI water and complete distillation to obtain 50.0 ml final volumn.
3.1.3 If distillate is turbid, filter through a 0.45 micron filter.

4.0 PHOTOMETRY

4.1 Manual

4.1.1 Using the 10 ppm phenol working solution, prepare the following standards in 16 x 125 mm tubes.

ml Phenol	DI Water	Conc. ug/ml (ppm)
0	10.0	0.00
0.1	9.9	0.10
0.25	9.75	0.25
0.5	9.5	0.50
10.0	0.0	1.00

For unknowns, use 10.0 ml.

4.1.2 Add 0.2 ml of buffer solution to standards and unknowns and mix. pH should be 10 +/- 0.2.

4.1.3 Add 0.2 ml of 4-Aminoantipyrine solution and mix.

4.1.4 Add 0.2 ml of Potassium Ferricyanide and mix.

4.1.5 Allow 15 minutes for color development and determine absorbance at 510 nm.

4.1.6 Report unknowns-- ug/ml (ppm). If outside calibration, dilute and reanalyze.

4.2 Automated Determination

4.2.1 Prepare the following standards:
2.0 ppm; 1.0 ppm; 0.5 ppm; 0.1 ppm; 0.05 ppm and 0.0 ppm.

4.2.2 Open phenol method from computer memory (ref. Lachat Quik-Chem S.O.P)

4.2.3 Build and load tray with standards, QC samples and samples to be analyzed Start tray..

4.2.4 Correlation of calibration should be greater than 0.99. If sample falls outside calibration, dilute and rerun.

5.0 CHLOROFORM EXTRACTION METHOD

5.1 From the distillation procedure above, place 100 ml of distillate in a 125 ml separatory funnel. For this extraction procedure, the sample should contain less than 25 ug phenols.

5.2 To the sample add 10 ml of buffer solution and mix pH should be 10 +/- 0.2.

5.3 Add 3.0 ml of 4-Aminoantipyrine solution and mix.

5.4 Add 3.0 ml of potassium ferricyanide and mix.

5.5 Allow to sit for 3.0 minutes; then extract with 10 ml of chloroform. Shake the funnel at least 10 times, let chloroform settle, shake again 10 times, let chloroform settle to bottom.

5.7 Filter extract through filter paper. Do not add more chloroform.

5.8 Determine the absorbance of samples and standards against a blank at 460 nm. If the concentration exceeds calibration, dilute and reanalyze.

6.0 CALCULATION

6.1 Prepare a calibration curve using 5 standards. Use the blank to zero UV.

6.2 Conc. (mg/L or mg/kg) = $\frac{ug\ in\ extract\ x\ dilution}{ml\ or\ g\ used}$

6.3 Report the following detection limits:

Distillation:	Water 0.05 ug/ml (ppm)
	Soil 0.50 ug/g (ppm)
Chloroform extraction:	Water 0.005 ug/ml (ppm)

7.0 QUALITY CONTROL

7.1 Analyze one blank, duplicate, spike and spike duplicate per batch or every 10 samples. Redistill if the recovery is not 75-125 and RPD < 15. Duplicate RPD <15.

7.2 Verify calibration with independent check standard immediately after initial calibration and every 15 samples. The standard must be within 10% of the true value, if not, correct the problem and reanalyze effected samples.

SULFIDE, ACID-SOLUBLE AND REACTIVE

1.1.0 REFERENCE

1.1 SW-846, Method 9030 B
1.2 EPA 600, Method 376.2
1.3 Standard Methods, Method 4500-S2-D
1.4 SW-846 Chapter 7
1.5 SW-846, Method 9034

2.0 REAGENTS

2.1 Zinc Acetate (0.5M), dissolve 110 g zinc acetate in 200 ml DI water, add 1 ml conc. HCL, dilute to 1 liter.
2.2 Sodium Hydroxide (1.25N), dissolve 50 g NaOH in 1 liter DI water.
2.3 Sulfuric Acid (18N), slowly add 500 ml conc. H_2SO_4 to 500 ml of DI water.
2.4 Starch Indicator, commercial.
2.5 Potassium Iodide (app. 0.025N), dissolve 25 g KI in 700 ml DI water, add 3.2 g iodine, mix well, add 2.0 ml of 6N HCl, dilute to 1 liter and standardize with sodium thiosulfate.
2.6 Sodium Thiosulfate (0.025N), dissolve 6.205 g sodium thiosulfate in 500 ml DI water, add 9.0 ml 1N NaOH, mix, dilute to 1 liter.

$$\text{Normality Iodine} = \frac{\text{ml titrant x normality titrant}}{\text{sample size in ml}}$$

2.7 Sulfide Stock Solution, prepare fresh, pH must be between 9 and 11, use sodium sulfide nonanhydride. Purge solution with helium. Standardize this solution daily using the iodometric titration method.
2.8 Amine-sulfuric Acid, dissolve 2.7 g N,N-dimethyl-p-phenylenediamine oxalate in cold mixture of 5.0 ml conc. sulfuric acid and 2.0 ml DI water; cool; dilute to 10.0 ml. Do not use if discolored. Dilute this to 1L with 1:1 sulfuric acid. Store in dark glass bottle; solution should be clear.
2.8.1 Automated Stock Reagent - in a 500 ml volumetric dissolve 1.0 g N,N-dimethyl-p-phenylenediamine dihydrochloride in 500 ml of 6 M hydrochloric acid. Discard when solution appears dark.
2.8.2 Prepare working diamine/Hcl reagent by diluting 200 ml of stock diamine reagent (2.8.1) to 1 L with DI water.
2.9 Ferric Chloride, dissolve 100 g $FeCl_3 \cdot 6H_2O$ in 40 ml DI water.
2.9.1 Automated Ferric Chloride Reagent (0.1 N) - in a 500 ml volumetric

dissolve 13.5 g of ferric chloride hexahydrate in 500 ml of 6 N hydrochloric acid.

2.10 Diammonium Hydrogen Phosphate, dissolve 200 g (NH4)2HPO4 in 400 ml DI water.

2.11 Phenylarsine oxide, 0.025 N, purchase from commercial source.

3.0 PROCEDURE

3.1 Distillation, Acid-Soluble:

3.1.1 Prior to disillation determine how much sulfuric acid is needed to obtain a pH less than 1 by placing 10 ml or 10 g of sample diluted to 40 ml with reagent water, check pH, while stirring slowly add conc. H2SO4. Perform test in a fume hood. CAUTION: samples may release toxic gases. Fill each scrubber with 2.0 ml of 0.5 M zinc acetate solution, 1.0 ml of 37% formaldehyde and 22 ml reagent water. Place 50 ml of sample or 10 g in 40 ml reagent water in the boiling tube, assemble and seal distillation apparatus, start nitrogen purge at about 25 ml/min. (app.10 psig) for 10 minutes, using dropping funnel add calculated amount of acid plus 10 ml additional. Note: solids must be broken up to allow for adequate agitation.

3.1.2 Set the following conditions: rate1=15 C/min; temp1=70 C;time1=1.5 hrs.

3.1.3 Start program, when at 70 C open stopcock of dropping funnel to add conc. sulfuric acid at app. 1 ml/min. Heat for 90 additional minutes. When cool remove both scrubbers, turn off nitrogen, combine scrubbers, minimize agitation and titrate immediately. .

3.2 Distillation, Reactivity:

3.2.1 Place 10 g of sample diluted to 250 ml in a one liter boiling flask, add 50.0 ml of 0.25 N NaOH to scrubber, connect nitrogen gas to distillation setup. Turn gas on and set flow to 60 ml, allow to purge for 1 to 2 minutes.

3.2.2 Add 250 ml of 0.2 N H_2SO_4 to addition funnel, open stopcock and add to boiling flask for final conc. of 0.1 N.

3.2.3 Start timer, begin stirring solution being sure not to create a vortex, after 30 minutes close off nitrogen and determine concentration of sulfide per p. 3.3.

3.3 Titration:

3.3.1 Pipet a known amount (app. 65 ml) of standardized iodine solution, 0.025N, into a 500 ml flask, bring volume to 100 ml with reagent water. NOTE: For reactivity titrations determine the amount of acid required to bring the pH to 2.0 by titrating a very small aliquot of scrubber, add back

aliquot and add calculated amount of HCl to iodine in flask. If not reactive test add 2.0 ml of 6N HCl, add a known volume of scrubber solution being careful not to discharge color. Shake well prior to transfer. If any visible precipitate is left in the scrubber tube rinse with known amount of standardized iodine, 1 ml 6N HCl and reagent water. Record total volume of iodine and sample.

3.3.2 Titrate the solution using 0.025N sodium thiosulfate or 0.025 N phenylarsine oxide until the amber color fades to yellow, add enough starch indicator to turn solution dark blue, continue titration until blue color disappears. Record total volume of titrant used.

3.3.3 Calculate concentration of sulfide as follows:

$$\text{Sulfide (mg/kg,mg/L)} = \frac{(\text{ml I2} \times \text{N I2}) - (\text{ml titrant} \times \text{N titrant}) \times 16.03}{\text{sample wt in kg or volume in liters}}$$

3.4 Colorimetric, Methylene Blue (includes Automated Method)

3.4.1 Prepare intermediate sulfide calibration standard by diluting the 1000 ppm sulfide stock standard to 10 ppm: i.e. 1 ml to 100 ml in DI water (pH>9). Purge solution with helium.

3.4.2 Prepare calibration standards by diluting the intermediate standard as follows:

ml intermediate std.	vol. (ml)	conc. (ppm)
0.0	7.5	0.00
0.1	7.4	0.10
0.25	7.25	0.25
0.5	7.0	0.5
1.0	6.5	1.0
2.0	5.5	2.0

3.4.3 Develop color by adding 0.5 ml Amine-sulfuric acid reagent and 0.15 ml FeCl3 solution. Mix by inverting once. Wait 3 to 5 minutes.

NOTE: If zinc acetate was used as a preservative, wait 10 minutes before reading.

3.4.3.1 For the automated method open the "Sulfide" method, build and load the tray with standards, samples and QC, start tray.

3.4.3.2 Correlation of calibration must be >0.99. Dilute if sample exceeds highest calibration standard.

3.4.4 Add 1.85 ml diammonium hydrogen phosphate; wait 10 minutes.

3.4.5 Read standards and samples at 664 nm using a 1 cm cell.

3.4.6 Multiply by dilution if needed.

4.0 QUALITY CONTROL

4.1 Minimize exposure of samples and standards to air. Prepare fresh. Purging with helium helps to minimize oxidation of sulfide.

4.2 Run control standards with each batch or group of 20 samples. Recovery must be 85-115%. NOTE: % recovery > 20 required for reactive sufide.

4.3 A duplicate, spike and spike duplicate must be brought through distillation process with an RPD < 15.

4.4 Total sulfides is defined as acid-soluble fraction for purposes of this procedure.

4.5 Minimum detectable limit (colorimetric) is 0.1 mg/L for water and 0.2 mg/L for solids. By titration 1.0 mg/l or mg/kg.

TCLP EXTRACTION PROCEDURE

1.0 REFERENCE
- 1.1 SW-846, Third Edition, Method 1311
- 1.2 Water Extraction Test (WET)

2.0 REAGENTS
- 2.1 Extraction fluid #1: Add 114.0 ml glacial acetic acid to 500 ml water. Add 128.60 ml 10N NaOH and dilute to 20L pH should be 4.93 +/-0.05. Record.
- 2.2 Extraction fluid #2: Dilute 11.4 ml glacial acetic acid to 2 liters with water. pH should be 2.88 +/- 0.05. Record.
- 2.3 Glacial Acetic Acid, ACS reagent grade.
- 2.4 Hydrochloric Acid (IN), ACS reagent grade, 83 ml conc. to 1 L DI water.
- 2.5 Sodium Hydroxide 10N ACS reagent grade, 400 g NaOH to 1 L DI water.

3.0 PROCEDURE
- 3.1 Determine the percent solids in the sample. (If sample visibly will yield no liquid do not continue this test.)
  - 3.1.1 Pre-weigh filter and collection vessel, assemble filtration apparatus, weigh out sample (100 g minimum) and record. Allow slury to settle or centrifuge.
  - 3.1.2 Quantitatively transfer sample to filter holder. (Both phases if applicable). Gradually apply about 10 psi up to maximum of 50 psi. If liquid fails to pass for 2 minutes, then stop filtration.
  - 3.1.3 Determine weight of liquid filtrant by subtracting initial filter and collection vessel weight. Determine weight of solid phase by subtracting weight of liquid from total sample weight. Record.
  - 3.1.4 Calculate percent solids as follows:

$$\% \text{ solid} = \frac{\text{weight of solid in grams (100)}}{\text{total weight of waste in grams}}$$

*Filter only sample must pass through 1 filter in 2 minutes or less; contain < 0.5% solid.

  - 3.1.5 If percent solids is greater or equal to 0.5 then evaluate its particle size. Particle size reduction is required if any narrowest dimension is greater than 1 cm (see supervisor).
  - 3.1.6 To determine the proper extraction fluid, add 5 g of solid to 96.5 ml water in a 500 ml beaker.Stir for five minutes and record the pH. If the pH is less than 5.0, us extraction fluid #1. If the pH is greater than 5.0, add 3.5 ml 1N Hcl, slurry briefly, cover the beaker with a watch glass, and heat to

50 deg C for 10 minutes. Cool and record the pH. If the pH is < 5.0, use fluid #1, if greater than 5.0 use fluid #2. Note that the zero headspace extraction uses only fluid #1.

3.1.7 Check ZHE after every extraction for leaks, use 50 psi for 1 hour or submerge and check for bubbles. Record.

3.2 Volatiles Not Involved

3.2.1 If the sample contains less than 0.5% solids, use the filtered liquid as the TCLP extract.

3.2.2 Prepare solids phase by cutting/grinding if necessary and determine the amount of extraction fluid needed as follows:

$$\text{Wt of fluid} = \frac{(20)(\text{percent solids})(\text{wt of sample})}{100}$$

3.2.3 Add the solid sample and the extraction fluid, close extraction vessel tightly, and place in rotation device (30 + 2 rpm) for 18 + 2 hours. Room temperature should be 23 + 2°C. Record.

3.2.4 After the extraction period, filter the extract through a clean 0.6 um filter.

3.2.5 Add previously separated liquid, if sample was multiphasic.

3.2.6 Record pH of TCLP extract. Aliquots for metals analysis must be acidified with HNO3 to pH <2.0.

3.2.7 TCLP extracts should be now prepared and analyzed according to applicable methods.

3.3 Volatiles Involved

3.3.1 Use the ZHE for volatiles only. Charge ZHE only once, do not open until final extract is collected.

3.3.2 If the sample contains less than 0.5% solids the liquid phase after filtering through 0.6 to 0.8 µm filter is the TCLP extract.

3.3.3 Determine weight of extraction fluid #1 needed as follows:

$$\text{Wt sample to charge ZHE} = \frac{25}{\text{percent solids}} \times 100$$

3.3.4 Weigh the ZHE unit. After size reduction, if reduction, transfer the entire sample into unit, seal, and tighten fittings. Do not attach collection device.

3.3.5 Slowly apply 1-10 psi to expell headspace. At first appearance of liquid close valve and stop applying pressure.

3.3.6 Attach evacuated, pre-weighed collection container to outlet, open valve and begin pressure. Increase in 10 psi increments to 50 psi. Weigh the ZHE unit. Record.

3.3.7 The liquid phase may be analyzed directly. Store at 4°C.

3.3.8 Determine weight of extraction fluid #1 needed as follows:

$$\text{wt. extraction fluid} = \frac{(20)(\text{percent solids})(\text{wt. of sample})}{100}$$

3.3.9 Attach line from extraction fluid resevoir to liquid inlet valve on ZHE. Preflush line to remove air. Release pressure on ZHE piston using gas valve, open liquid inlet valve, transfer extraction fluid until correct amount has been added. Close liquid valve. Rapidly expel headspace. Repressurize to 10 psi. Check that valves are closed.

3.3.10 Place ZHE in rotation device and rotate at 30 + 2 rpm for 18 +/- 2 hours. Check that device is still pressurized. If not, repeat extraction.

3.3.11 If original sample contained no liquid phase, this liquid is the TCLP extract. If sample contained an initial liquid phase, it should be added to this extract. Store at 4°C. Analyze according to applicable method.

4.0 QUALITY CONTROL

4.1 A blank using the same extraction fluid used for the sample must be analyzed for every 20 extraction on glass containers.

4.2 One matrix spike should be analyzed per batch; add after filtration of TCLP extract but before preservation.

4.3 A Blank using the same extraction fluid used for the sample must be analyzed for every 10th extraction when applied to ZHE's.

4.4 All ZHE's, glassware, and extraction vessels must be heated for a minimum of 2 hours @ 250° F before use with another sample.

4.5 Any containers stained, etched, or scratched by a sample during rotation should be disposed and replaced.

4.6 All filters used for the determination of metals must be acid washed with 1 N nitric acid and rinsed three times with DI water.

APPENDIX
WATER EXTRACTION TEST

1. Extraction Solution - 0.2 M sodium citrate at pH of 5.0 =/- 0.1 by titrating analytical grade citric acid in DI water with 4.0 N sodium hydroxide. For hexavalent chromium use DI water.
2. Type i - if waste or other material is a millable solid the sample shall pass directly or be milled to pass through a No. 10 (two millimeter) standard sieve before extraction.
   Type ii - if waste is a filterable mixture of liquid and solids in which solids are 0.5% or greater by weight, separate by using a 0.45 um filter, record initial filtrate volume, sieve or mill solids to pass No. 10 sieve, extract solids with 1:10 ratio liquid to solid, combine initial liquid and filtrate extract
   Type iii - if waste is nonfilterable and nonmillable or oily or tarry it shall be analyzed as received.
   Note: if waste or other material is a liquid containing less than 0.5 % by weight of undissolved solids it shall not be subject to WET procedure but shall be analyzed directly. The waste is hazardous if any total concentration exceeds the TTLC value. If the total concentration is less than the TTLC but exceeds the STLC in mg/l the waste shall be filtered through 0.45 um membrane filter, the solids discarded and the filtrate analyzed directly.
3. Extract as follows: rinse extraction container with 1:1 by volume nitric acid, if extract is to be analyzed for organics a glass container must be used. If type ii use 50 g, place in suitable clean polyethylene or glass container, add 500 ml of extraction fluid, fit with covered air scrubbers extending into solution and shake vigorously with nitrogen gas for 15 minutes. If sample is to be analyzed for any volatile organics add sample after dearation with nitrogen. Quickly seal and place in rotary extractor. Extract for 48 hours between 20 and 40 C. A blank must be carried through entire procedure with each batch. Filter through medium porosity prefilter and then a 0.45 um membrane filter.
4. Metals - transfer filtered extracts to clean polyethylene bottle and acidify with nitric acid to 5% by volume.
   Organics - transfer filtered extract to clean glass bottles, do not acidify, if not analyzed within 24 hours freeze.
   Fluoride - transfer extract to clean polyethylene bottles, do not acidify if not analyzed within 24 hours freeze.

TOTAL ORGANIC CARBON

1.0 REFERENCE
- 1.1 SW-846, Method 9060A.
- 1.2 EPA 600, Method 415.1
- 1.3 Walkley-Black Method for Soils

2.0 REAGENTS
- 2.1 Organic Carbon Standard, dissolve 0.2125 g of Potassium Hydrogen Phthalate (primary standard grade) in 100 ml DI water (conc.=1000 ppm).
- 2.2 Inorganic Carbon Standard, dissolve 0.3500 g Sodium Bicarbonate and 0.4418 g of Sodium Carbonate in 100 ml DI water (conc.= 1000 ppm total).
- 2.3 Sulfuric Acid Solution, dilute concentrated H2SO4 with DI water, ratio of 1:1.
- 2.4 Check Standard, EPA Standard or equivalent, commercial.
- 2.5 Certified Organic Carbon Standard (1000 ppm), commercial.

3.0 PROCEDURE
- 3.1 Instrument Set-Up
  - 3.1.1 Turn "ON" oxygen gas, initiate start-up, check Rotometer for flow.
  - 3.1.2 Allow instrument to reach operating temperature. Unit is operational when "Ready" light is illuminated.

- 3.2 ANALYSIS
  - 3.2.1 Water
    - 3.2.1.1 Inject water blanks until less than 3.0 ppm with an SD < 5%.
    - 3.2.1.2 Using Organic and Inorganic standards prepare at least three calibration standards as follows:

Vol. Stock Std. (ml)	Final Vol. (ml)	Conc. (ppm)
0.1 ml	50	2.0
0.5 ml	50	10
2.5 ml	50	50
5.0 ml	50	100

    - 3.2.1.3 Verify calibration using certified standard from second source. The standard must be read within 10% of the true value.
    - 3.2.1.4 Daily check instrument using mid-point calibration standard. If the daily standard is not within 10% of the true value, recalibrate.
    - 3.2.1.5 Transfer about 5.0 ml of sample to autosampler. Samples with

significant particle content should be well mixed before removal.

3.2.1.6 Set instrument to add 50 ul sulfuric acid and sparge for 4 minutes.

3.2.1.7 Analyze samples in quadruplicate, and report average. If concentration exceeds calibration, dilute with DI water and reanalyze. Record dilution.

3.2.2 SOILS

3.2.2.1 Weigh out about 0.1 g homogenized soil and transfer to a COD reactor tube, cap, place in block digestor for 2 hours at 150 C. Analyze using COD SOP # 8.

3.2.2.2 Using the TOC standard prepare a five-point calibration using 1.0, 10, 20, 40 and 60 mg/kg standards.

3.2.2.3 Weigh 0.015 g TOC soil control and place in reactor tube/reactor. Take result divided by weight times 2.33, confirm result is within certified acceptable range.

3.3 INSTRUMENT SHUT-DOWN

3.3.1 Initiate "Finish" command from Stand-By Option (Main Menu).

3.3.2 After cool, initiate "Stand-By", turn "OFF" screen. Do not turn off main power switch.

4.0 CALCULATIONS

4.1 Record "MN" value from printer. Correct for dilution (MN x dilution).

5.0 QUALITY CONTROL

5.1 WATER - A blank, duplicate (every 10 samples), spike and spike duplicate are required on each batch or every 20 samples. Spike recovery must be 75-125% with an RPD <20. Duplicate RPD <20.

5.2 SOILS - A blank, sample duplicate and laboratory control is required every batch or every 20 samples. RPD < 20. Lab Control must be within supplier certified range of acceptance.

METHOD 8260 B

VOLATILE ORGANIC ANALYSIS BY GC/MS

1.0 SCOPE AND APPLICATION

This method is suitable for the determination of volatile organics, boiling points less than 200 C, in water and various solid matrices including oils. The estimated quantitation limit will vary with each compound but is about 0.002 mg/L or 0.002 ug/g. For applicable compounds with retention times see chromatogram at end of procedure. This procedure is restricted to use by analysts **experienced** in purge and trap GC/MS and skilled in the interpretation of mass spectra.

2.0 SUMMARY OF METHOD

Volatiles are purged from the matrix using an inert gas, trapped on a solid sorbent, thermally desorbed and quantitated by capillary GC/MS. Identification of targets is accomplished by comparing their mass spectra with the electron impact of spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard.

3.0 INTERFERENCES

3.1 Interferences usually consist of elevated SW-846 Method 8260 blanks due to volatiles used in the lab or carryover from a previous sample that was very concentrated. Do not blank subtract. Prep lab personnel are not allowed in volatile lab.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph/Mass Spectrometer - Hewlett Packard 5971 or 5972 MSD. Hewlett Packard 5890-II programmable gas chromatograph. HP Chemstation and Enviroquant software used to control, aquire and process data.
Column: DB-VRX 60 m x 0.25mm, 1.4um film thickness.

4.2 Purge and Trap Device - Tekmar LSC 3000/ALS 2016 or Dynatech PTA30 with Teckmar 3000. Systems must be able to heat soils to 40 C and purge 5.0 ml or 5.0 g of sample.

4.3 Syringes, Hamilton or equivalent, 10 ul, 25 ul, 50 ul, 100 ul, 500 ul, 1 ml and 5 ml.

4.4 Balance, top-loading, 0.1 g accuracy, commercial source.

4.5 Glassware, class A, 10 ml and 100 ml.

5.0 REAGENTS

METHOD 8260 B

5.1 Methanol - Purge and trap grade or equivalent, commercial source.

5.2 Reagent Water - Deionized or distilled water in which no interferences are noted at a level above the practical quantitation limit (PQL) for any parameter of interest.

5.3 Stock VOA Standard - Stock standard solutions (200 ug/ml) may be prepared on a weight/volume basis in methanol using pure standard material, or may be purchased as certified solutions commercially (Ultra DWM-580 or equivalent). Store in amber bottle with a teflon-lined screw cap at -10 C or less. All certified standards are good for 6 months. Second Source Calibration Verification - NSI C-350, 200 ug/ml.

5.4 Working VOA Standard - Dilute 500 ul of stock VOA standard to 2.0 ml in MeOH for a 50 ug/ml standard. Store at - 10 C or less, good for one week.

5.5 Synthetic Soil, Sea Sand, precleaned, commercial source.

5.6 Working Internal and Surrogate Standard - Obtain a 2000 ug/ml internal standard (Ultra STM-341N, chlorobenzene-d5, 1,4-difluorobenzene, 1,4-dichlorobenzene-d4 and pentafluorobenzene) and a 2000 ug/ml surrogate mix (Accustandard M8260A/B-SS, 4-bromofluorobenzene, dibromofluoromethane and toluene-d8). For the working IS/SS standard for the 2016 system dilute 30 ul each to 2.0 ml with MeOH for a 30 ug/ml each standard. Add 5.0 ul to 5.0 ml water sample or to 5.0 g soil for a 30 ug/L or 30 ug/kg solution. For the PTA-30 dilute 2.0 ml of stock to 26.65 ml MeOH for a 150 ug/ml solution. Place in autosample standard syringe, 1.0 ul in 5 ml or 5 g equals 30 ug/L or 30 ug/kg each.

5.7 4-Bromofluorobenzene (BFB) standard, Accustandard CLP-004-100X, 2500 ug/ml or equivalent - dilute 20 ul to 2.0 ml with methanol for a 25 ug/ml standard, use 10 ul per 5.0 ml water for purging (50 ug/L) or inject 2.0 ul for 50 ng.

5.8 Safety - Treat all chemicals as potential carcinogens. Minimize exposure, wear gloves and prepare all standards in a hood, if possible. MSDS's located in Client Services.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Aqueous samples should be collected in duplicate using pre-cleaned VOA vials with teflon-lined septa screw cap. Preserve to pH < 2 with HCl. Refrigerate at 4 +/- 2 C. Analyze within 14 days. (Non-preserved samples must be analyzed within 7 days).

6.2 All glassware should be Class A, clean per SOP # 32.

7.0 PROCEDURE

7.1 TUNING - The GC/MS system must be tuned to meet the Bromofluorobenzene (BFB) requirements every 12 hrs. Inject 2.0 ul of 25 ug/ml BFB working

METHOD 8260 B

standard onto the GC column and analyze using a 35C to 110 C temperature program ramping at 8 C /min. Display the scan of interest and generate a list of the masses and their percent relative abundances. Compare to the requirements stated below and if the requirements are met, generate a copy of the relevant data. No calibration or sample analysis may begin until a successful tune has been generated.

NOTE: Purging a 50 ug/L BFB standard is acceptable for tuning.

MASS	**ION ABUNDANCE CRITERIA**
50	15 to 40 % of mass 95
75	30 to 60 % of mass 95
95	BASE PEAK, 100% RELATIVE ABUNDANCE
96	5 to 9 % of mass 95
173	less than 2% of mass 174
174	greater than 50 % of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% OF MASS 174
177	5 to 9% of mass 176

7.2 INITIAL CALIBRATION - A 5 point calibration curve must be generated for every target compound and surrogates. The levels required for initial calibration are 10 ppb, 20 ppb, 50 ppb, 100 ppb and 200 ppb for all surrogates and target compounds. Prepare as follows using the 50 ug/ml working standards:

IS (ul)	SS (ul 50 ug/ml)	VOA Std. (ul of 50 ug/ml)	final vol. (ml)	conc. (ug/L)
5	1	1	5	10
5	2	2	5	20
5	5	5	5	50
5	10	10	5	100
5	20	20	5	200

Each of the five analyses should contain 30 ug/l of each internal standard.

METHOD 8260 B

Analyze each standard and each sample under the same conditions i.e.,: Purge Time: 11.0 minutes; Trap Temp: <30 C; Desorb Time: 2.0 minutes; Desorb Temp: 225 C; Bake Time: 10 minutes; Bake Temp. 250 C; Jacketed Heater: soils to 40 C. Set GC as follows: Init. Temp : 45° C; Time 1: 6.0 minutes; Rate 1: 10.0 C/minute; Final Temp : 190 C; Final Time: 2.0 minutes.

The average response factor and relative standard deviation are calculated for each of the five concentrations, and the 5 point analysis is evaluated for the following:

7.2.1 The RSD of all target compounds must be less than 15%.

7.2.2 The 6 CCC compounds (1,1-Dichloroethene, Chloroform, Vinyl Chloride,1,2-Dichloropropane, Toluene, and Ethylbenzene) must have a relative standard deviation of less than 30 %.

7.2.3 The 5 SPCC compounds (Chloromethane,1,1-Dichloroethane, Bromoform,1,1,2,2-Tetrachloroethane, and Chlorobenzene) must have an minimum relative response factor as follows:

Chloromethane and 1,1 DCA	0.1
Bromoform	0.1
Chlorobenzene and TCA	0.3

If the 5 point calibration curve fails to meet these criteria, corrective actions should be taken and the calibration curve re-analyzed. All target compounds are quantitated using linear-regression, the correlation coefficient must be equal or greater than 0.99 or recalibrate. When using regression do not force the line through zero and do not incorporate a zero concentration standard as a sixth point. Verify initial calibration using a 50 ppb second source standard (NSI C-350). Results must be within 20 % or recalibrate.

7.2.4 Calculate response factor as follows:

RF = (area of ion target x conc. int. std) / (area of ion int. std. x conc. target)

7.2.5 Calculate final concentration as follows:

Conc. (ug/L or ug/kg) = (area target x conc. IS x dilution factor) / (areas IS x RF)

7.3 DAILY CALIBRATION - After a satisfactory initial calibration curve has been established and verified, the system must be checked every 12 hours using a daily tune standard (50 ng BFB) and a continuing calibration verification standard containing 50 ppb of each target analyte (5 ul of working VOA standard in 5 ml water). After quantitation of the standard, the CCC and SPCC compounds are checked against the 5 point calibration for the criteria described below.

METHOD 8260 B

7.3.1 The 6 CCC compounds must have a relative percent difference of less than or equal to 20 as compared to the 5 point calibration.

7.3.2 The response factor for the 5 SPCC compounds must be as specified in p. 7.2.3.

7.3.3 The % D ((true calibration check conc.-measured conc.))(100)/true calibration check conc. of all targets must be equal or less than 20 except for oxygenated compounds which must be equal or less than 40.

7.3.4 Evaluate the internal standard responses and the retention times. If the RT of any internal standard changes by more than 30 seconds or the area of any internal standard changes by a factor of two, correct problem and reanalyze all affected samples. (see chromatogram at end of SOP)

7.3.5 If the daily standard does not meet the above criteria, re-prepare the 50 ug/ml solution and re-analyze. If this does not correct the problem, a new 5 point calibration curve must be generated. All data relevant to the 5 point calibration standard and the daily calibration standard should be maintained in the QC data book.

7.4 METHOD BLANK - Before analysis of each batch of samples, a method blank must be analyzed using 5 ml DI water or 5 g synthetic soil. Fill a 5.0 ml gas tight syringe with DI water, add 5.0 microliters of the IS/SS solution containing 30 ug/ml of each to 5.0 ml of DI water. Fill position on autosampler to be purged. After quantitation, the method blank should not contain any of the analytes of interest at a level greater than the PQL. If any analyte is present at a level greater than the PQL, a new blank must be analyzed until the system is free from any interferences. Surrogate recovery in the blank must conform to at least the criteria listed below:

	WATER	SOIL
4-BFB	86 - 115	74 - 121
DBFM	86 - 118	80 - 120
Toluene-d8	88 - 110	81 - 117
1,2-DCA-d4	80 - 120	80 - 120

If the recovery of the surrogates do not meet the specified criteria, the blank must be reanalyzed. All data relevant to the blank should be filed with other QC data for documentation.

7.5 SAMPLE ANALYSIS - Allow samples to reach room temperature before analysis. When using the Tekmar 2016 autosampler place 5.0 ml of the sample into the 5.0 ml gas tight syringe and transfer to open position on the 2016

METHOD 8260 B

autosampler. Add 5.0 ul of the IS/SS solution containing 30 ug/ml of each internal standard and surrogate standard to the unknown and purge the sample as described above. For soils weigh a 5.0 g aliquot, place in autosampler, place 5.0 ml of DI water in a 5 ml gas-tight syringe, add 5.0 ul of the 30 ug/ml IS/SS internal standard and surrogate standard, add to soil position in autosampler, heat to 40 C and purge as before. When using the Dynatrap PTA-30 autosampler, fill the standard syringe with 150 ug/ml internal standard and surrogates. 1.0 ul is automatically added to each sample for a final concentration of 30 ppb each. The recovery of the internal standards and surrogate standards are calculated and compared to the limits specified above. If recovery is not within the specified range, the sample must be reanalyzed. If reanalysis of the sample does not correct the situation, the system should be examined and action taken to correct the situation. If the concentration of any analyte is above the working range of the instrument (i.e., 200 ug/L), an appropriate dilution of the sample must be analyzed. Use a second unopened VOA vial to repeat analysis or prepare a dilution. The operator's experience with both this method and with the instrument should weigh heavily on the dismissal or acceptance of the data generated. Check the pH of the sample with indicator paper. Note in logbook to nearest whole pH. Dilute water samples by injecting appropriate amount into 5 ml gas-tight syringe partially filled with DI water. For water-miscible liquids prepare a 50 X dilution by injecting 100 ul into a 5 ml DI water in a 5 ml syringe. For soils, dilutions may be made by reducing the amount purged i.e. min. of 1 g or extracting 5 g with 5.0 ml methanol and injecting 100 ul into 5 ml DI water in a 5 ml gas-tight syringe for a 50 X dilution. Do not inject more than 100 ul of methanol per 5 ml water.

7.6 Determine dilution factor as if 1 g was purged instead of 5 g the enter a dilution factor of 5. Enter in dilution field of LIMS which will multiply the integrated result times that factor. For methanol extractions when using 5 g sample to 5 ml methanol. If needed, determine dilution factor for solids as follows: 5 / ml MeOH purged.

7.7 All calculatioms must be performed by the analyst and indicated on the worklist prior to entry into the LIMS.

8.0 QUALITY CONTROL

8.1 MATRIX SPIKE / MATRIX SPIKE DUPLICATE/LCS - A matrix spike, matrix spike duplicate and LCS should be analyzed per batch, not to exceed 20 samples of a given matrix. Recovery ranges for matrix spikes shall be within statistically derived limits. After analysis of the original sample, 5.0 ml (water) or 5.0 g (soil) of the sample is reanalyzed after spiking with 5 ul of working VOA standard (50

METHOD 8260 B

ppb) of the target compounds. Check % recovery and confirm that the following are within limits below: 1,1-dichloroethane, trichloroethene, benzene, toluene, and chlorobenzene. If the sample required dilution on the original run in order to bring all analyte concentrations into the calibration range of the instrument, the same dilution should be analyzed for the matrix spike and matrix spike duplicate. The percent recovery and RPD of the matrix spike and spike duplicate compounds is calculated and compared to the QC limits specified:

Compound	% Recovery Water	RPD	% Recovery Soil	RPD Soil
1,1 - DCE	61 - 145	0 - 14	59 - 172	0 - 22
TCE	71 - 120	0 - 14	62 - 137	0 - 24
Benzene	76 - 127	0 - 11	66 - 142	0 - 21
Toluene	76 - 125	0 - 13	59 - 139	0 - 21
Chlorobenzene	75 - 130	0 - 13	60 - 133	0 - 21

All relevant QC requirements as pertains to internal and surrogate standard recoveries is also evaluated. The amount of each of the matrix spike compounds present in the original sample should be subtracted from the values determined by the matrix spike and matrix spike duplicate analyses. The relative percent difference between the matrix spike and matrix spike duplicate is calculated as follows:

$$\frac{[\text{matrix spike}] - [\text{matrix spike duplicate}]}{[\text{matrix spike} + \text{matrix spike duplicate}/2]} * 100\%$$

For every batch, a 50 ppb LCS (laboratory control standard) using 5 ul of working VOA standard containing all target compounds in 5 ml DI water or 5 g synthetic soil must be analyzed. Determine % recovery for each analyte. Recovery must be 70 - 130 % or repeat all affected samples. If any IS/SS fails repeat all samples in the batch.

All QA/QC data pertaining to the calibration procedures (both the initial 5 point calibration curve and all daily standards), all method blanks, and all matrix spike/matrix spike duplicates should be filed in a separate QA/QC file for documentation and quick reference to any sample analyses to which they pertain. All QA/QC data should be approved by the GC/MS supervisor or senior analyst before sample analysis begins.

METHOD 8260 B

8.2 MDL's must be determined yearly per 40 CFR 136 Appendix A. For waters use a 0.002 ug/ml concentration and for soils use 0.005 ug/g. Calculate using the standard deviation of seven consecutive replicates, multiply std. deviation by 3.14. The result must be less than the reporting level.

8.3 Control charts will be used for trend analysis on the LCS, MS and MSD. These are generated monthly. Examples are attached.

9.0 REFERENCES

9.1 SW-846 Method 8260**B**, Rev.2, Jan 1995

10.0 CORRECTIVE ACTION

10.1 Each applicable section contains the required corrective action if specified criteria are outside limits.

10.2 Most problems may be corrected by changing traps, remaking a standard, performing column maintenance, etc. All maintanence is to be recorded in the maintenance log.

10.3 If routine maintenance does not correct the problem notify your supervisor immediately.

Quantitation Report

Data File : C:\HPCHEM\1\DATA\VS0709B.D
Acq Time : 10 Jul 97 9:04 am
Sample : CON CAL
Misc :
Quant Time: Jul 10 14:48 1997

Operator: HP-1
Inst : 5971 - In
Multiplr: 1.00

Method : C:\HPCHEM\1\METHODS\8260S.M
Title : 8260 VOLATILES
Last Update : Thu Jul 10 09:25:12 1997
Response via : Multiple Level Calibration

Internal Standards	R.T.	QIon	Response	Conc	Units		Dev(Min)
1) Pentafluorobenzene	13.62	168	216659	30.00	ug/L		0.00
26) 1,4-Difluorobenzene	14.84	114	366683	30.00	ug/L		0.00
42) Chlorobenzene-d5	19.31	119	90940	30.00	ug/L		-0.01
55) 1,4-Dichlorobenzene-d4	23.00	152	128657	30.00	ug/L		-0.01
System Monitoring Compounds							%Recovery
21) 1,2-Dichloroethane-d4	13.71	65	109739	30.61	ug/L		102.04%
22) Dibromofluoromethane	13.06	111	110063	30.19	ug/L		100.64%
38) Toluene-d8	17.34	98	376667	30.06	ug/L		100.19%
57) Bromofluorobenzene	20.98	95	127750	28.49	ug/L		94.95%
Target Compounds							Qvalue
2) Dichlorodifluoromethane	6.12	85	186330	59.23	ug/L		82
3) Chloromethane	14.54	50	376323	59.97	ug/L		95
4) Vinyl Chloride	6.94	62	253476	49.71	ug/L	#	1
5) Bromomethane	7.78	96	156688	51.68	ug/L		97
6) Chloroethane	8.04	64	190451	48.63	ug/L	#	81
7) Trichlorofluoromethane	9.08	101	202234	40.88	ug/L		96
8) Acetone	9.24	43	31561	34.49	ug/L		91
9) 1,1-Dichloroethene	10.01	96	187731	53.50	ug/L	#	75
10) Methylene Chloride	10.25	84	233754	56.48	ug/L	#	67
11) Carbon Disulfide	10.64	76	460524	52.03	ug/L		100
12) trans-1,2-Dichloroethene	11.33	61	387291	57.15	ug/L	#	80
13) Methyl-t-butyl ether	11.50	73	498449	52.12	ug/L		95
14) 1,1-Dichloroethane	11.71	63	430885	57.19	ug/L	#	98
15) 2-Butanone	12.38	43	426491	50.47	ug/L	#	75
16) Diisopropyl ether	12.38	45	854797	53.05	ug/L	#	85
17) cis-1,2-Dichloroethene	12.57	61	302936	56.80	ug/L	#	80
18) Bromochloromethane	12.82	130	125616	54.13	ug/L		95
19) Chloroform	12.88	83	368126	57.28	ug/L		99
20) 2,2-Dichloropropane	13.00	77	296867	54.47	ug/L		93
23) 1,2-Dichloroethane	13.83	62	246605	52.87	ug/L	#	95
24) 1,1,1-Trichloroethane	13.97	97	289060	55.23	ug/L	#	92
25) 1,1-Dichloropropene	14.22	75	318538	57.63	ug/L		97
27) Carbon Tetrachloride	14.49	117	234181	52.39	ug/L		97
28) Benzene	14.55	78	870746	53.34	ug/L		100
29) Dibromomethane	15.33	174	106441	50.78	ug/L	#	82
30) 1,2-Dichloropropane	15.37	63	233236	52.90	ug/L	#	87
31) Trichloroethene	15.43	130	226180	54.25	ug/L		96
32) 2-Chloro vinyl ether	15.37	63	233236	49.65	ug/L		89
33) Bromodichloromethane	15.50	129	24602	50.11	ug/L		84
34) cis-1,3-Dichloropropene	16.36	75	289025	48.93	ug/L		98
35) 4-Methyl-2-Pentanone	16.49	43	196686	49.46	ug/L	#	83
36) trans-1,3-Dichloropropene	16.94	75	226488	46.36	ug/L		97

(#) = qualifier out of range (m) = manual integration

Quantitation Report

Data File : C:\HPCHEM\1\DATA\VS0709B.D
Acq Time : 10 Jul 97 9:04 am
Sample : CON CAL
Misc :
Quant Time: Jul 10 14:48 1997

Operator: HP-1
Inst : 5971 - In
Multiplr: 1.00

Method : C:\HPCHEM\1\METHODS\8260S.M
Title : 8260 VOLATILES
Last Update : Thu Jul 10 09:25:12 1997
Response via : Multiple Level Calibration

Compound	R.T.	QIon	Response	Conc Unit	Qvalue
37) 1,1,2-Trichloroethane	17.18	97	139522	50.70 ug/L	98
39) Toluene	17.44	91	834146	55.24 ug/L	100
40) 1,3-Dichloropropane	17.49	76	267446	49.95 ug/L #	72
41) 2-Hexanone	17.67	43	116832	45.17 ug/L #	82
43) Dibromochloromethane	17.89	129	152153	49.87 ug/L	97
44) 1,2-Dibromoethane	18.23	107	145042	50.82 ug/L	97
45) Tetrachloroethene	18.44	166	197234	55.13 ug/L	91
46) 1,1,1,2-Tetrachloroethane	19.26	131	164922	53.47 ug/L	97
47) Chlorobenzene	19.37	112	511402	54.28 ug/L	99
48) Ethylbenzene	19.61	91	870818	54.35 ug/L	98
49) m,p-Xylene	19.86	91	1285446	110.39 ug/L	97
50) Bromoform	20.07	173	78002	45.02 ug/L	99
51) Styrene	20.32	104	535099	51.77 ug/L	100
52) o-Xylene	20.42	91	663923	56.39 ug/L	96
53) 1,1,2,2-Tetrachloroethane	19.25	133	155921	52.91 ug/L	1
54) 1,2,3-Trichloropropane	20.61	110	45211	48.93 ug/L	41
56) Isopropylbenzene	20.91	105	878370	53.58 ug/L	97
58) Bromobenzene	21.30	77	319968	52.14 ug/L #	82
59) Propylbenzene	21.55	91	991631	52.99 ug/L #	97
60) 2-Chlorotoluene	21.72	91	562722	54.05 ug/L #	96
61) 4-Chlorotoluene	21.82	91	536284	51.16 ug/L	97
62) 1,3,5-Trimethylbenzene	21.96	105	652608	53.82 ug/L	98
63) t-Butylbenzene	22.43	119	609721	55.11 ug/L	98
64) 1,2,4-Trimethylbenzene	22.59	105	637973	51.86 ug/L	100
65) sec-Butylbenzene	22.78	105	968017	55.79 ug/L	94
66) 1,3-Dichlorobenzene	22.94	146	343549	50.95 ug/L	99
67) 1,4-Dichlorobenzene	23.05	146	333969	49.49 ug/L	100
68) 1,2-Dichlorobenzene	23.69	146	311233	50.69 ug/L	100
69) p-Isopropyltoluene	22.43	119	609721	55.03 ug/L #	60
70) -Butylbenzene	23.81	91	676473	51.85 ug/L	92
72) 1,2,4-Trichlorobenzene	27.57	180	159775	43.19 ug/L	98
73) Naphthalene	28.16	128	359466	44.32 ug/L	100
74) Hexachlorobutadiene	28.27	225	125832	51.70 ug/L	96
75) 1,2,3-Trichlorobenzene	28.63	180	139291	43.00 ug/L	96

(#) = qualifier out of range (m) = manual integration

Quantitation Report

Data File : C:\HPCHEM\1\DATA\VS0709B.D
Acq Time : 10 Jul 97 9:04 am
Sample : CON CAL
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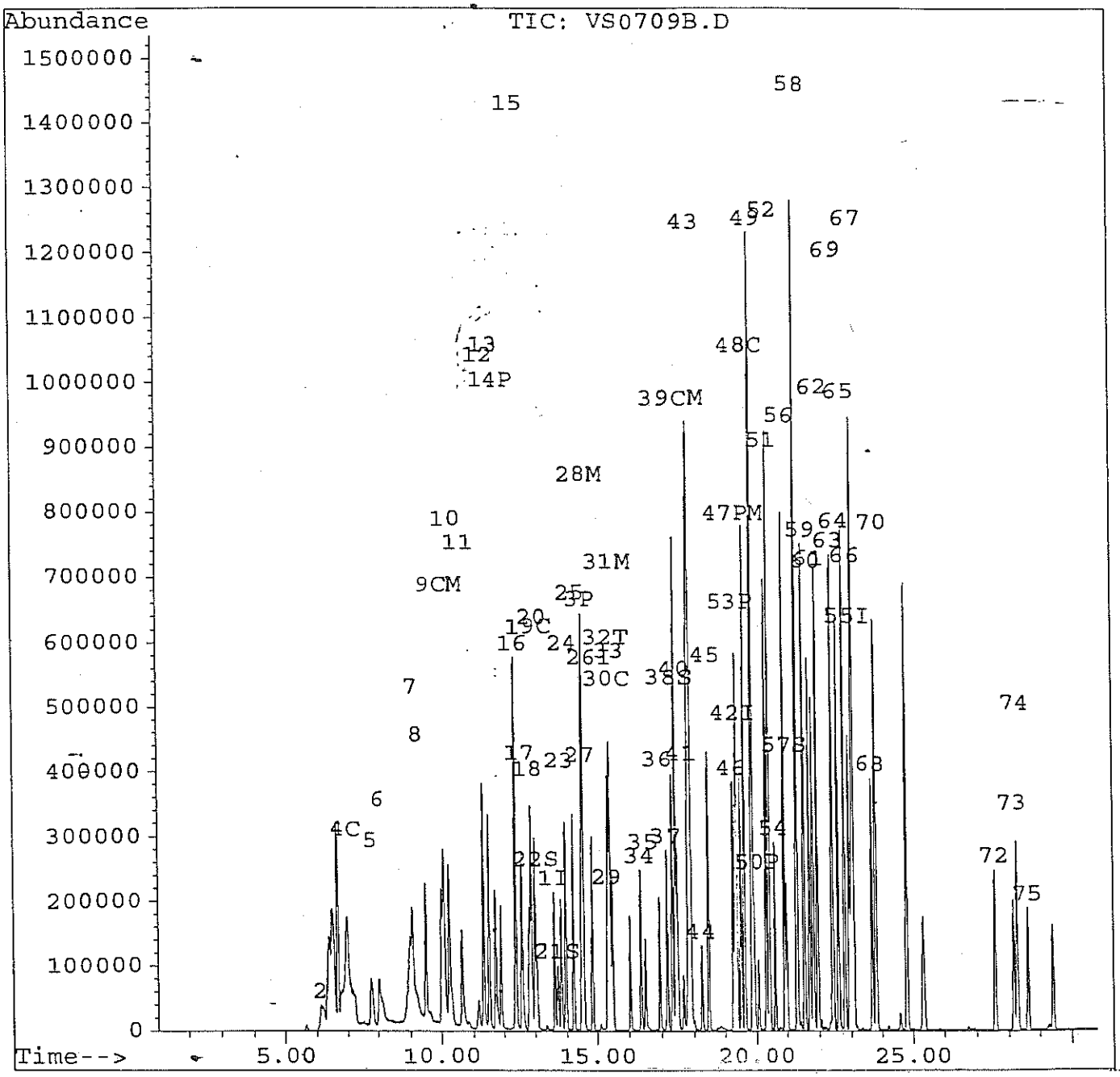
Title : 8260 VOLATILES
Last Update : Thu Jul 10 09:25:12 1997
Response via : Multiple Level Calibration



METHOD 8151

EXTRACTION & DERIVATIZATION, CHLORINATED HERBICIDES

1.0 REFERENCES

1.1 SW-846, 3rd Edition, Method 8151 A

1.2 Standard Methods for the examination of water and wastewater, 17th Ed., Method 6640

2.0 REAGENTS

2.1 Sulfuric acid, reagent grade, commercial source, (1:3) - Slowly add 25 ml conc. sulfuric acid to 75 ml DI water.

2.2 Hydrochloric acid, conc., reagent grade, commercial source.

2.3 Potassium hydroxide, reagent grade, commercial source, prepare 37% solution by dissolving 37 g KOH pellets in 100 ml DI.

2.4 Acetone - Pesticide grade or equivalent, commercial.

2.5 Diethyl ether - Pesticide grade or equivalent commercial.

2.6 Isooctane, methanol - Pesticide grade or equivalent, commercial.

2.7 Methylene Chloride - Pesticide grade or equivalent, commercial.

2.8 Acidified, anhydrous, Sodium sulfate - Heat granular sodium sulfate in an oven for 4 hours at 400 ° C. Acidify by slurrying 100 g sodium sulfate with just enough diethyl ether to cover the solid; add 0.1 ml concentrated sulfuric acid and mix thoroughly. Remove the ether by evaporation. Mix 1 g with 5 ml water and measure the pH. The pH must be less than 4. Store at 130 C.

2.9 Diazald - Aldrich Chemical Co.

2.10 Surrogate, 2,4-dichlorophenyl acetic acid standard, 5.0 ug/ml, spike with 1.0 ml.

2.11 Herbicide Spike, free acids, spike with 1.0 ml.

3.0 EXTRACTION

3.1 Soils, Sediment or Other Solids

3.1.1 Using concentrated HCl, adjust the pH of 30 g, dry weight, of a well mixed sample to < 2 . Dry to free flowing mix using acidified sodium sulfate.

3.1.2 Add 1.0 ml surrogate standard and if needed 1.0 ml spike.

3.1.3 Add 100 ml methylene chloride/acetone (1:1) to beaker, sonicate for 3 minutes at full power, pulse at 50% duty cycle. Allow solids to settle. Transfer organic layer to beaker. Extract twice more using same process. Combine all extracts. Filter through acidified sodium sulfate into a 500 ml flask. Add 10 g acidified sodium sulfate and periodically shake. Dry for at least 2 hours.

METHOD 8151

3.1.4 Transfer to K-D and reduce volume to 5 ml. Add 5 ml of 37 % aqueous potassium hydroxide to the extract, add boiling chip, reflux the mixture for 2 hours in a 60-65 C water bath. Allow to cool.

3.1.5 Transfer the hydrolyzed aqeous solution to a separatory funnel and extract three times with 100 ml portions of methylene chloride. Discard. The herbicides are in the basic water phase.

3.1.8 Adjust the water phase to a pH less than 2 with cold sulfuric acid (1:3), mix well, extract with 40 ml diethyl ether and twice with 20 ml portions of ether. Combine extracts, filter through pre-rised and acidified sodium sulfate. Collect the dried extract in a flask containing 10 g acidified anhydrous sodium sulfate. Periodically shake, allow to dry at least 2 hours.

3.1.9 Quantitatively transfer to K-D and concentrate the sample using a 3-ball Snyder column. Concentrate to approximately 4 ml of ether. Transfer to diazomethane generator.

3.2 Aqueous Samples

3.2.1 Using a 1 L graduated cylinder, measure approximately but accurately 1 L of sample. Place the sample into a 2 L separatory funnel. Add 250 g of NaCl, shake until dissolved. Add 1 ml surrogate and if needed 1 ml spike. Add 17 ml of 6 N NaOH, shake, check that pH is greater than 12 or add more NaOH. Let sit for at least 2 hours with periodic shaking.

3.2.2 Rinse the sample bottle with 60 ml of methylene chloride and pour the rinsate into the separatory funnel.

3.2.3 Shake the funnel for 2 minutes with periodic venting. Allow the layers to separate for at least 10 minutes. Mechanically break any emulsions. Discard the organic layer.

3.2.4 Repeat the extraction two times using 60 ml methylene chloride each time. Discard organic layer. Add 17 ml cold 12 N sulfuric acid to the hydrolyzed sample, shake. Confirm pH is less than 2 or add more acid.

3.2.5 Add 120 ml diethyl ether, shake well for 2 minutes with venting. Allow layers to separate for at least 10 minutes. Mechanically break any emulsion. Collect the ether phase in a flask containing 10 g of acidified anhydrous sodium sulfate. Vigorously shake extract with drying agent. Return water phase to separatory funnel and repeat extract twice with 60 ml portions of ether. Combine all ether portions. Allow to remain in contact with drying agent for at least 2 hours.

3.2.7 Remove the sodium sulfate by filtering through acidified glass wool. Transfer to K-D, rinse flask with ether and add to K-D, add boiling chips,

METHOD 8151

concentrate to about 0.5 ml, rinse snyder with about 0.2 ml ether, add 1.0 ml isooctane and 0.5 ml methanol, dilute to 4 ml with ether. Transfer to diazomethane generator.

4.0 DERIVATIZATION

4.1 Ground Water/ Soils

4.1.1 Prepare the diazomethane by adding 4.5 ml ether to a clean, dry, unscratched 40 ml vial with a septum top.

4.1.2 After adding 0.15 g Diazald to a 12 x 75 mm tube, place the tube into the larger vial which contains the ether. Using a syringe, add 0.5 ml water to the tube containing the Diazald. Seal the vial.

4.1.3 Place the apparatus in an ice bath under a hood. DO NOT ALLOW EXPOSURE TO GENERATED TOXIC GASES!

4.1.4 Using a syringe, puncture the septum and slowly add 0.5 ml 5 N NaOH dropwise into the reagent tube. Gas evolution should be obvious. **NOTE:** Pressure in the vial could eject the plunger from the syringe!

4.1.5 Remove the syringe and check the system for leaks. (A drop of water placed at the puncture point will verify re-sealing of the vial.)

4.1.6 Allow the reaction 20 minutes to complete; then carefully - under the hood - remove the vial cap. There should be some pressure within the vial.

4.1.7 The ether should be yellow from the diazomethane present. Remove the inner reaction tube. If using more than one vial, combine the generated diazomethane.

4.1.8 Add 2.0 ml of the diazomethane reagent to the ether extract from steps 3.1.16 and 3.2.11.

4.1.9 Submerge all generator components in water to remove all traces of reactants.

4.1.10 The derivatization should proceed for 30 minutes after which the ether is removed under a gentle stream of nitrogen. The solution may be warmed slightly.

4.1.11 Once the ether is removed - near dryness - 10 ml toluene is added.

4.1.12 The samples are ready for analysis.

4.2 Wastewater (2,4-D, 2,4,5-T, silvex only)

4.2.1 The extract from 3.2.11 is combined with 0.5 ml toluene and concentrated to 0.5 ml using a K-D apparatus or nitrogen blowdown.

4.2.2 Add 0.5 ml boron trifluoride reagent. Heat at 50° C for 30 minutes.

4.2.3 Cool and add acidified sodium sulfate solution so that the upper toluene layer can be easily removed. Shake vigorously.

METHOD 8151

4.2.4 After phase separation remove the toluene.

4.2.5 A Pasteur pipet is plugged with glass wool and packed with 1.5 cm magnesia-silica gel over-layered with 2.0 cm acidified sodium sulfate.

4.3.6 Pipet the toluene from 4.2.4 onto the top of the column. Rinse the receiver from step 4.2.3 with small (0.5 ml) volumes of toluene and add these to the top of the column once the column has drained below the top of the solid. The final column eluent volume should be 2.0 ml. Dilute to 5.0 ml with toluene.

5.0 QA/QC

5.1 Water samples must be extracted within 7 days, soils within 14 days.

5.2 All glassware and associated items should be acid washed or low recoveries may result.

APPENDIX C
CHAIN OF CUSTODY FORM

W. Z. Baumgartner & Associates, Inc.
ENGINEERS AND ENVIRONMENTAL CONSULTANTS
500 WILSON PIKE CIRCLE • SUITE 208 • BRENTWOOD, TN 37027
P. O. BOX 786 (37024) • 615-373-1572 • FAX: 615-370-9292
E-MAIL: WZBINC@AOL.COM

CHAIN OF CUSTODY

Page ____ of ____

PART I - Client:

Proj. No: | Location:

PART II - Container Information.

	Preservative						Plastic Containers							Glass Containers									
Code	A	B	C	D	E	F	1	2	3	4	5	6	7	8	9	10	11	12	13	14			
Quantity of Sample Containers Shipped	H_2SO_4	HNO_3	HCL	$Na_2S_2O_3$	NaOH	Unpreserved	125 ml HDPE	250 ml HDPE	500 ml HDPE	1l HDPE	1gal HDPE	Poly Bag	5 gal. Bucket	40 mil Vial	4 oz Jar	8 oz Jar	16 oz Jar	1l Clear	1l Amber	Trip Blank			Notes

Preservative added: ☐ By Contract Laboratory ☐ By WZB Laboratory ☐ In Field

PART III - Sample Information

Sample Matrix Codes

DW	Drinking Water	SL	Sludge
WW	Wastewater	SO	Soil
GW	Groundwater	SR	Shredder Residue
SW	Storm Water		
SU	Surface Water	BL	Blank
OI	Oil		

Sample I.D.	Date	Time	Matrix	Comp.	Grab	Preserv./Container Code	Requested Analysis	Total No. of Containers

Sampler's Name (Print):

PART IV - Chain of Custody

	Relinquished By: (Signature)	Date:	Time:	Received By: (Signature)
1				
2				
3				
4				
Received for Laboratory By: (Signature)				Custody Seals Intact: ☐ Yes ☐ No Temperature: (°C) _____

WZB

APPENDIX D

PARAMETER LIST

TABLE D-1 - SOIL PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Antimony	6010B	10
Arsenic	6010B	1.0
Beryllium	6010B	1.0
Cadmium	6010B	1.0
Chromium	6010B	1.0
Copper	6010B	1.0
Lead	6010B	1.0
Mercury	7471A	0.1
Nickel	6010B	1.0
Selenium	6010B	1.0
Silver	6010B	1.0
Thallium	6010B	1.0
Zinc	6010B	10
Cyanide	9012A	2.0
Sulfide	9030A	1.0
Chloride	9056,9251	1.0
Phenols	9066	0.5

TABLE D-1 (CONTINUED) - SOIL PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Aroclor 1016	8082	0.033
Aroclor 1221	8082	0.067
Aroclor 1232	8082	0.033
Aroclor 1242	8082	0.033
Aroclor 1248	8082	0.033
Aroclor 1254	8082	0.033
Aroclor 1260	8082	0.033
Aldrin	8081A	0.0017
a-BHC	8081A	0.0017
b-BHC	8081A	0.0017
d-BHC	8081A	0.0017
g-BHC, Lindane	8081A	0.0017
Chlordane	8081A	0.0017
4,4'-DDD	8081A	0.0033
4,4'-DDE	8081A	0.0033
4,4'DDT	8081A	0.0033
Dieldrin	8081A	0.0033
Endosulfan I	8081A	0.0017
Endosulfan II	8081A	0.0033
Endosulfan sulfate	8081A	0.0033

TABLE D-1 (CONTINUED) - SOIL PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Endrin	8081A	0.0033
Endrin aldehyde	8081A	0.0033
Heptachlor	8081A	0.0017
Heptachlor epoxide	8081A	0.0017
Methoxychlor	8081A	0.0167
Toxaphene	8081A	0.167

TABLE D-1 (CONTINUED) - SOIL PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Acetone	8260B	0.010
Acetonitrile	8260B	0.005
Acrolein	8260B	0.010
Acrylonitrile	8260B	0.010
Allyl chloride	8260B	0.010
Benzene	8260B	0.002
Bromodichloromethane	8260B	0.002
Bromoform	8260B	0.002
Bromomethane	8260B	0.002
2-Butanone	8260B	0.010
Carbon disulfide	8260B	0.002
Carbon tetrachloride	8260B	0.002
Chlorobenzene	8260B	0.002
Chloroethane	8260B	0.002
Chloroform	8260B	0.002
Chloromethane	8260B	0.002
Chloroprene	8260B	0.005
1,2-Dibromo-3-chloropropane	8260B	0.002
Dibromochloromethane	8260B	0.002
1,2-Dibromoethane	8260B	0.002
Dibromomethane	8260B	0.002

TABLE D-1 (CONTINUED) - SOIL PARAMETERS

PARAMETER	METHOD	PQL mg/kg
1,4-Dichloro-2-butene	8260B	0.002
1,2-Dichlorobenzene	8260B	0.002
1,3-Dichlorobenzene	8260B	0.002
1,4-Dichlorobenzene	8260B	0.002
Dichlorodifluoromethane	8260B	0.002
1,1-Dichloroethane	8260B	0.002
1,2-Dichloroethane	8260B	0.002
1,1-Dichloroethene	8260B	0.002
1,2-Dichloroethene (total)	8260B	0.002
1,2-Dichloropropane	8260B	0.002
cis-1,3-Dichloropropene	8260B	0.002
trans-1,3-Dichloropropene	8260B	0.002
1,4-Dioxane	8260B	0.10
Ethylbenzene	8260B	0.002
Ethyl methacrylate	8260B	0.010
Hexachlorobutadiene	8260B	0.002
2-Hexanone	8260B	0.010
Iodomethane	8260B	0.002
Isobutyl alcohol	8260B	0.010
Methacrylonitrile	8260B	0.005
Methyl methacrylate	8260B	0.005

TABLE D-1 (CONTINUED) - SOIL PARAMETERS

PARAMETER	METHOD	PQL mg/kg
4-Methyl-2-pentanone	8260B	0.010
Methylene chloride	8260B	0.002
Pentachloroethane	8260B	0.002
Propionitrile	8260B	0.005
Styrene	8260B	0.002
1,1,1,2-Tetrachloroethane	8260B	0.002
1,1,2,2-Tetrachloroethane	8260B	0.002
Tetrachloroethene	8260B	0.002
Toluene	8260B	0.002
1,2,4-Trichlorobenzene	8260B	0.002
1,1,1-Trichloroethane	8260B	0.002
1,1,2-Trichloroethane	8260B	0.002
Trichloroethene	8260B	0.002
Trichlorofluoromethane	8260B	0.002
1,2,3-Trichloropropane	8260B	0.002
Vinyl acetate	8260B	0.010
Vinyl chloride	8260B	0.002
Xylenes	8260B	0.002

TABLE D-1 (CONTINUED) - SOIL PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Acenaphthene	8270C	0.333
Acenaphthylene	8270C	0.333
Acetophenone	8270C	0.333
2-Acetylaminofluorene	8270C	1.670
4-Aminobiphenyl	8270C	1.670
Aniline	8270C	0.825
Anthracene	8270C	0.333
Aramite	8270C	0.333
Benzo(a)anthracene	8270C	0.333
Benzo(a)pyrene	8270C	0.333
Benzo(b)fluoranthene	8270C	0.333
Benzo(g,h,i)perylene	8270C	0.333
Benzo(k)fluoranthene	8270C	0.333
Benzyl Alcohol	8270C	1.67
4-Bromophenylphenylether	8270C	0.333
Butylbenzylphthalate	8270C	0.333
4-Chloro-3-methylphenol	8270C	0.333
4-Chloroaniline	8270C	0.333
Chlorbenzilate	8270C	1.67
bis(2-Chloroethoxy)methane	8270C	0.333
bis(2-Chloroethyl)ether	8270C	0.333

TABLE D-1 (CONTINUED) - SOIL PARAMETERS

PARAMETER	METHOD	PQL mg/kg
bis(2-Chloroisopropyl)ether	8270C	0.333
Bis(2-ethylhexyl)phthalate	8270C	0.333
2-Chloronaphthalene	8270C	0.333
2-Chlorophenol	8270C	0.333
4-Chlorophenylphenylether	8270C	0.333
Chrysene	8270C	0.333
Diallate	8270C	1.67
Dibenzofuran	8270C	0.333
Dibenz(a,h)anthracene	8270C	0.333
3,3'-Dichlorobenzidine	8270C	0.666
2,4-Dichlorophenol	8270C	0.333
2,6-Dichlorophenol	8270C	1.67
Diethylphthalate	8270C	0.333
Dimethoate	8270C	1.670
p-Dimethylaminoazobenzene	8270C	1.670
3,3'-Dimethylbenzidine	8270C	0.666
7,12-Dimethylbenz[a]anthracene	8270C	1.67
2,4-Dimethylphenol	8270C	0.333
Dimethylphthalate	8270C	0.333
a,a-Dimethylphenethylamine	8270C	0.333
Di-n-butylphthalate	8270C	0.333

TABLE D-1 (CONTINUED) - SOIL PARAMETERS

PARAMETER	METHOD	PQL mg/kg
4,6-Dinitro-2-methylphenol	8270C	0.825
1,3-Dinitrobenzene	8270C	1.67
1,2-Dinitrobenzene	8270C	0.333
2,4-Dinitrophenol	8270C	0.825
2,4-Dinitrotoluene	8270C	0.333
2,6-Dinitrotoluene	8270C	0.333
Di-n-octylphthalate	8270C	0.333
Dinoseb	8270C	0.333
Diphenylamine	8270C	1.67
Disulfoton	8270C	0.333
Ethylmethane sulfonate	8270C	1.67
Famphur	8270C	1.67
Fluoranthene	8270C	0.333
Fluorene	8270C	0.333
Hexachlorobenzene	8270C	0.333
Hexachlorocyclopentadiene	8270C	0.333
Hexachloroethane	8270C	0.333
Hexachlorophene	8270C	0.333
Hexachloropropene	8270C	1.67
Indeno(1,2,3-cd)pyrene	8270C	0.333
Isodrin	8270C	1.670

TABLE D-1 (CONTINUED) - SOIL PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Isophorone	8270C	0.333
Isosafrole	8270C	1.670
Methapyrilene	8270C	1.670
3-Methylcholanthrene	8270C	1.670
Methylmethanesulfonate	8270C	1.67
2-Methylnaphthalene	8270C	0.333
2-Methylphenol	8270C	0.333
3-Methylphenol	8270C	0.333
m,p-Methylphenol	8270C	0.333
Naphthalene	8270C	0.333
1,4-Naphthaquinone	8270C	1.67
1-Naphthylamine	8270C	1.67
2-Naphthylamine	8270C	1.67
2-Nitroaniline	8270C	0.825
3-Nitroaniline	8270C	0.825
4-Nitroaniline	8270C	0.825
Nitrobenzene	8270C	0.333
5-Nitro-o-toluidine	8270C	1.67
2-Nitrophenol	8270C	0.333
4-Nitrophenol	8270C	0.825
4-Nitroquinoline N-oxide	8270C	0.333

TABLE D-1 (CONTINUED) - SOIL PARAMETERS

PARAMETER	METHOD	PQL mg/kg
N-nitrosodibutylamine	8270C	1.67
N-nitrosodiethylamine	8270C	1.67
N-nitrosodi-n-propylamine	8270C	0.333
N-nitrosodiphenylamine	8270C	0.333
N-nitrosodimethylamine	8270C	0.333
N-nitrosomethylethylamine	8270C	1.67
N-nitrosomorpholine	8270C	0.333
N-nitrosopiperidine	8270C	1.67
N-nitrosopyrrolidine	8270C	1.67
Pentachlorobenzene	8270C	1.670
Pentachloronitrobenzene	8270C	1.670
Pentachlorophenol	8270C	0.825
Phenacetin	8270C	1.670
Phenanthrene	8270C	0.333
Phenol	8270C	0.333
1,4-Phenylenediamine	8270C	0.333
Phorate	8270C	0.333
2-Picoline	8270C	0.333
Pronamide	8270C	1.670
Pyrene	8270C	0.333
Pyridine	8270C	0.333

TABLE D-1 (CONTINUED) - SOIL PARAMETERS

PARAMETER	METHOD	PQL. mg/kg
Safrole	8270C	1.670
1,2,4,5-Tetrachlorobenzene	8270C	1.670
2,3,4,6-Tetrachlorophenol	8270C	1.670
Tetraethylpyrophosphate	8270C	0.333
Thionazine	8270C	1.670
o-Toluidine	8270C	0.333
1,2,4-Trichlorobenzene	8270C	0.333
2,4,5-Trichlorophenol	8270C	0.825
2,4,6-Trichlorophenol	8270C	0.333
o,o,o-Triethylphosphorothioate	8270C	1.670
1,3,5-Trinitrobenzene	8270C	1.670

TABLE D-2 - SEDIMENT PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Antimony	6010B	10
Arsenic	6010B	1.0
Beryllium	6010B	1.0
Cadmium	6010B	1.0
Chromium	6010B	1.0
Copper	6010B	1.0
Lead	6010B	1.0
Mercury	7471A	0.1
Nickel	6010B	1.0
Selenium	6010B	1.0
Silver	6010B	1.0
Thallium	6010B	1.0
Zinc	6010B	10
TOC	Walkly Black	30

TABLE D-3 - COMPOSITE FLUFF SAMPLES PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Antimony	6010B	10
Arsenic	6010B	1.0
Beryllium	6010B	1.0
Cadmium	6010B	1.0
Chromium	6010B	1.0
Copper	6010B	1.0
Lead	6010B	1.0
Mercury	7471A	0.1
Nickel	6010B	1.0
Selenium	6010B	1.0
Silver	6010B	1.0
Thallium	6010B	1.0
Zinc	6010B	10
Cyanide	9012A	2.0
Sulfide	9030A	1.0
Chloride	9056,9251	1.0
Phenols	9066	0.5
Reactivity	s.8.3 SW-846	
Corrosivity	1110	6.35 mm/yr
Flash Point	1010	
Paint Filter	9095A	

TABLE D-3 (CONTINUED) - COMPOSITE FLUFF SAMPLES PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Aroclor 1016	8082	0.033
Aroclor 1221	8082	0.067
Aroclor 1232	8082	0.033
Aroclor 1242	8082	0.033
Aroclor 1248	8082	0.033
Aroclor 1254	8082	0.033
Aroclor 1260	8082	0.033
Aldrin	8081A	0.0017
a-BHC	8081A	0.0017
b-BHC	8081A	0.0017
d-BHC	8081A	0.0017
g-BHC, Lindane	8081A	0.0017
Chlordane	8081A	0.0017
4,4'-DDD	8081A	0.0033
4,4'-DDE	8081A	0.0033
4,4'DDT	8081A	0.0033
Dieldrin	8081A	0.0033
Endosulfan I	8081A	0.0017
Endosulfan II	8081A	0.0033
Endosulfan sulfate	8081A	0.0033

TABLE D-3 (CONTINUED) - COMPOSITE FLUFF SAMPLES PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Endrin	8081A	0.0033
Endrin aldehyde	8081A	0.0033
Heptachlor	8081A	0.0017
Heptachlor epoxide	8081A	0.0017
Methoxychlor	8081A	0.0167
Toxaphene	8081A	0.167

TABLE D-3 (CONTINUED) - COMPOSITE FLUFF SAMPLES PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Acetone	8260B	0.010
Acetonitrile	8260B	0.005
Acrolein	8260B	0.010
Acrylonitrile	8260B	0.010
Allyl chloride	8260B	0.010
Benzene	8260B	0.002
Bromodichloromethane	8260B	0.002
Bromoform	8260B	0.002
Bromomethane	8260B	0.002
2-Butanone	8260B	0.010
Carbon disulfide	8260B	0.002
Carbon tetrachloride	8260B	0.002
Chlorobenzene	8260B	0.002
Chloroethane	8260B	0.002
Chloroform	8260B	0.002
Chloromethane	8260B	0.002
Chloroprene	8260B	0.005
1,2-Dibromo-3-chloropropane	8260B	0.002
Dibromochloromethane	8260B	0.002
1,2-Dibromoethane	8260B	0.002
Dibromomethane	8260B	0.002

TABLE D-3 (CONTINUED) - COMPOSITE FLUFF SAMPLES PARAMETERS

PARAMETER	METHOD	PQL mg/kg
1,4-Dichloro-2-butene	8260B	0.002
1,2-Dichlorobenzene	8260B	0.002
1,3-Dichlorobenzene	8260B	0.002
1,4-Dichlorobenzene	8260B	0.002
Dichlorodifluoromethane	8260B	0.002
1,1-Dichloroethane	8260B	0.002
1,2-Dichloroethane	8260B	0.002
1,1-Dichloroethene	8260B	0.002
1,2-Dichloroethene (total)	8260B	0.002
1,2-Dichloropropane	8260B	0.002
cis-1,3-Dichloropropene	8260B	0.002
trans-1,3-Dichloropropene	8260B	0.002
1,4-Dioxane	8260B	0.10
Ethylbenzene	8260B	0.002
Ethyl methacrylate	8260B	0.010
Hexachlorobutadiene	8260B	0.002
2-Hexanone	8260B	0.010
Iodomethane	8260B	0.002
Isobutyl alcohol	8260B	0.010
Methacrylonitrile	8260B	0.005
Methyl methacrylate	8260B	0.005

TABLE D-3 (CONTINUED) - COMPOSITE FLUFF SAMPLES PARAMETERS

PARAMETER	METHOD	PQL mg/kg
4-Methyl-2-pentanone	8260B	0.010
Methylene chloride	8260B	0.002
Pentachloroethane	8260B	0.002
Propionitrile	8260B	0.005
Styrene	8260B	0.002
1,1,1,2-Tetrachloroethane	8260B	0.002
1,1,2,2-Tetrachloroethane	8260B	0.002
Tetrachloroethene	8260B	0.002
Toluene	8260B	0.002
1,2,4-Trichlorobenzene	8260B	0.002
1,1,1-Trichloroethane	8260B	0.002
1,1,2-Trichloroethane	8260B	0.002
Trichloroethene	8260B	0.002
Trichlorofluoromethane	8260B	0.002
1,2,3-Trichloropropane	8260B	0.002
Vinyl acetate	8260B	0.010
Vinyl chloride	8260B	0.002
Xylenes	8260B	0.002

TABLE D-3 (CONTINUED) - COMPOSITE FLUFF SAMPLES PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Acenaphthene	8270C	0.333
Acenaphthylene	8270C	0.333
Acetophenone	8270C	0.333
2-Acetylaminofluorene	8270C	1.670
4-Aminobiphenyl	8270C	1.670
Aniline	8270C	0.825
Anthracene	8270C	0.333
Aramite	8270C	0.333
Benzo(a)anthracene	8270C	0.333
Benzo(a)pyrene	8270C	0.333
Benzo(b)fluoranthene	8270C	0.333
Benzo(g,h,i)perylene	8270C	0.333
Benzo(k)fluoranthene	8270C	0.333
Benzyl Alcohol	8270C	1.67
4-Bromophenylphenylether	8270C	0.333
Butylbenzylphthalate	8270C	0.333
4-Chloro-3-methylphenol	8270C	0.333
4-Chloroaniline	8270C	0.333
Chlorbenzilate	8270C	1.67
bis(2-Chloroethoxy)methane	8270C	0.333
bis(2-Chloroethyl)ether	8270C	0.333

TABLE D-3 (CONTINUED) - COMPOSITE FLUFF SAMPLES PARAMETERS

PARAMETER	METHOD	PQL mg/kg
bis(2-Chloroisopropyl)ether	8270C	0.333
Bis(2-ethylhexyl)phthalate	8270C	0.333
2-Chloronaphthalene	8270C	0.333
2-Chlorophenol	8270C	0.333
4-Chlorophenylphenylether	8270C	0.333
Chrysene	8270C	0.333
Diallate	8270C	1.67
Dibenzofuran	8270C	0.333
Dibenz(a,h)anthracene	8270C	0.333
3,3'-Dichlorobenzidine	8270C	0.666
2,4-Dichlorophenol	8270C	0.333
2,6-Dichlorophenol	8270C	1.67
Diethylphthalate	8270C	0.333
Dimethoate	8270C	1.670
p-Dimethylaminoazobenzene	8270C	1.670
3,3'-Dimethylbenzidine	8270C	0.666
7,12-Dimethylbenz[a]anthracene	8270C	1.67
2,4-Dimethylphenol	8270C	0.333
Dimethylphthalate	8270C	0.333
a,a-Dimethylphenethylamine	8270C	0.333
Di-n-butylphthalate	8270C	0.333

TABLE D-3 (CONTINUED) - COMPOSITE FLUFF SAMPLES PARAMETERS

PARAMETER	METHOD	PQL mg/kg
4,6-Dinitro-2-methylphenol	8270C	0.825
1,3-Dinitrobenzene	8270C	1.67
1,2-Dinitrobenzene	8270C	0.333
2,4-Dinitrophenol	8270C	0.825
2,4-Dinitrotoluene	8270C	0.333
2,6-Dinitrotoluene	8270C	0.333
Di-n-octylphthalate	8270C	0.333
Dinoseb	8270C	0.333
Diphenylamine	8270C	1.67
Disulfoton	8270C	0.333
Ethylmethane sulfonate	8270C	1.67
Famphur	8270C	1.67
Fluoranthene	8270C	0.333
Fluorene	8270C	0.333
Hexachlorobenzene	8270C	0.333
Hexachlorocyclopentadiene	8270C	0.333
Hexachloroethane	8270C	0.333
Hexachlorophene	8270C	0.333
Hexachloropropene	8270C	1.67
Indeno(1,2,3-cd)pyrene	8270C	0.333
Isodrin	8270C	1.670

TABLE D-3 (CONTINUED) - COMPOSITE FLUFF SAMPLES PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Isophorone	8270C	0.333
Isosafrole	8270C	1.670
Methapyrilene	8270C	1.670
3-Methylcholanthrene	8270C	1.670
Methylmethanesulfonate	8270C	1.67
2-Methylnaphthalene	8270C	0.333
2-Methylphenol	8270C	0.333
3-Methylphenol	8270C	0.333
m,p-Methylphenol	8270C	0.333
Naphthalene	8270C	0.333
1,4-Naphthaquinone	8270C	1.67
1-Naphthylamine	8270C	1.67
2-Naphthylamine	8270C	1.67
2-Nitroaniline	8270C	0.825
3-Nitroaniline	8270C	0.825
4-Nitroaniline	8270C	0.825
Nitrobenzene	8270C	0.333
5-Nitro-o-toluidine	8270C	1.67
2-Nitrophenol	8270C	0.333
4-Nitrophenol	8270C	0.825
4-Nitroquinoline N-oxide	8270C	0.333

TABLE D-3 (CONTINUED) - COMPOSITE FLUFF SAMPLES PARAMETERS

PARAMETER	METHOD	PQL mg/kg
N-nitrosodibutylamine	8270C	1.67
N-nitrosodiethylamine	8270C	1.67
N-nitrosodi-n-propylamine	8270C	0.333
N-nitrosodiphenylamine	8270C	0.333
N-nitrosodimethylamine	8270C	0.333
N-nitrosomethylethylamine	8270C	1.67
N-nitrosomorpholine	8270C	0.333
N-nitrosopiperidine	8270C	1.67
N-nitrosopyrrolidine	8270C	1.67
Pentachlorobenzene	8270C	1.670
Pentachloronitrobenzene	8270C	1.670
Pentachlorophenol	8270C	0.825
Phenacetin	8270C	1.670
Phenanthrene	8270C	0.333
Phenol	8270C	0.333
1,4-Phenylenediamine	8270C	0.333
Phorate	8270C	0.333
2-Picoline	8270C	0.333
Pronamide	8270C	1.670
Pyrene	8270C	0.333
Pyridine	8270C	0.333

TABLE D-3 (CONTINUED) - COMPOSITE FLUFF SAMPLES PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Safrole	8270C	1.670
1,2,4,5-Tetrachlorobenzene	8270C	1.670
2,3,4,6-Tetrachlorophenol	8270C	1.670
Tetraethylpyrophosphate	8270C	0.333
Thionazine	8270C	1.670
o-Toluidine	8270C	0.333
1,2,4-Trichlorobenzene	8270C	0.333
2,4,5-Trichlorophenol	8270C	0.825
2,4,6-Trichlorophenol	8270C	0.333
o,o,o-Triethylphosphorothioate	8270C	1.670
1,3,5-Trinitrobenzene	8270C	1.670

TABLE D-4 - DISCRETE FLUFF SAMPLE PARAMETERS

PARAMETER	METHOD	PQL mg/kg
Cadmium (TCLP)	6010B/1311	1.0
Lead (TCLP)	6010B/1311	1.0
Aroclor 1016	8082	0.033
Aroclor 1221	8082	0.067
Aroclor 1232	8082	0.033
Aroclor 1242	8082	0.033
Aroclor 1248	8082	0.033
Aroclor 1254	8082	0.033
Aroclor 1260	8082	0.033

TABLE D-5 - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
Antimony	6010B	0.005
Arsenic	6010B	0.005
Beryllium	6010B	0.004
Cadmium	6010B	0.001
Chromium	6010B	0.005
Copper	6010B	0.010
Lead	6010B	0.003
Mercury	7470A	0.0002
Nickel	6010B	0.010
Selenium	6010B	0.005
Silver	6010B	0.005
Thallium	6010B	0.002
Zinc	6010B	0.020
Cyanide	9012A	0.010
Sulfide	9030A	0.100
Chloride	9056,9251	1.0
Phenols	9066	0.05

TABLE D-5 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
Aroclor 1016	8082	0.0005
Aroclor 1221	8082	0.0005
Aroclor 1232	8082	0.0005
Aroclor 1242	8082	0.0005
Aroclor 1248	8082	0.0005
Aroclor 1254	8082	0.0005
Aroclor 1260	8082	0.0005
Aldrin	8081A	0.00005
a-BHC	8081A	0.00005
b-BHC	8081A	0.00005
d-BHC	8081A	0.00005
g-BHC, Lindane	8081A	0.00005
Chlordane	8081A	0.00005
4,4'-DDD	8081A	0.0001
4,4'-DDE	8081A	0.0001
4,4'DDT	8081A	0.0001
Dieldrin	8081A	0.0001
Endosulfan I	8081A	0.00005
Endosulfan II	8081A	0.0001
Endosulfan sulfate	8081A	0.0001

TABLE D-5 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
Endrin	8081A	0.0001
Endrin aldehyde	8081A	0.0001
Heptachlor	8081A	0.0001
Heptachlor epoxide	8081A	0.00005
Methoxychlor	8081A	0.0001
Toxaphene	8081A	0.003

TABLE D-5 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
Acetone	8260B	0.010
Acetonitrile	8260B	0.005
Acrolein	8260B	0.010
Acrylonitrile	8260B	0.010
Allyl chloride	8260B	0.010
Benzene	8260B	0.002
Bromodichloromethane	8260B	0.002
Bromoform	8260B	0.002
Bromomethane	8260B	0.002
2-Butanone	8260B	0.002
Carbon disulfide	8260B	0.002
Carbon tetrachloride	8260B	0.002
Chlorobenzene	8260B	0.002
Chloroethane	8260B	0.002
Chloroform	8260B	0.002
Chloromethane	8260B	0.002
Chloroprene	8260B	0.005
1,2-Dibromo-3-chloropropane	8260B	0.002
Dibromochloromethane	8260B	0.002
1,2-Dibromoethane	8260B	0.002
Dibromomethane	8260B	0.002

TABLE D-5 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
1,4-Dichloro-2-butene	8260B	0.002
1,2-Dichlorobenzene	8260B	0.002
1,3-Dichlorobenzene	8260B	0.002
1,4-Dichlorobenzene	8260B	0.002
Dichlorodifluoromethane	8260B	0.002
1,1-Dichloroethane	8260B	0.002
1,2-Dichloroethane	8260B	0.002
1,1-Dichloroethene	8260B	0.002
1,2-Dichloroethene (total)	8260B	0.002
1,2-Dichloropropane	8260B	0.002
cis-1,3-Dichloropropene	8260B	0.002
trans-1,3-Dichloropropene	8260B	0.002
1,4-Dioxane	8260B	0.10
Ethylbenzene	8260B	0.002
Ethyl methacrylate	8260B	0.010
Hexachlorobutadiene	8260B	0.002
2-Hexanone	8260B	0.010
Iodomethane	8260B	0.002
Isobutyl alcohol	8260B	0.010
Methacrylonitrile	8260B	0.005
Methyl methacrylate	8260B	0.005

TABLE D-5 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
4-Methyl-2-pentanone	8260B	0.010
Methylene chloride	8260B	0.002
Pentachloroethane	8260B	0.002
Propionitrile	8260B	0.005
Styrene	8260B	0.002
1,1,1,2-Tetrachloroethane	8260B	0.002
1,1,2,2-Tetrachloroethane	8260B	0.002
Tetrachloroethene	8260B	0.002
Toluene	8260B	0.002
1,2,4-Trichlorobenzene	8260B	0.002
1,1,1-Trichloroethane	8260B	0.002
1,1,2-Trichloroethane	8260B	0.002
Trichloroethene	8260B	0.002
Trichlorofluoromethane	8260B	0.002
1,2,3-Trichloropropane	8260B	0.002
Vinyl acetate	8260B	0.002
Vinyl chloride	8260B	0.002
Xylenes	8260B	0.002

TABLE D-5 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
Acenaphthene	8270C	0.010
Acenaphthylene	8270C	0.01
Acetophenone	8270C	0.010
2-Acetylaminofluorene	8270C	0.050
4-Aminobiphenyl	8270C	0.050
Aniline	8270C	0.025
Anthracene	8270C	0.01
Aramite	8270C	0.01
Benzo(a)anthracene	8270C	0.01
Benzo(a)pyrene	8270C	0.010
Benzo(b)fluoranthene	8270C	0.01
Benzo(g,h,i)perylene	8270C	0.01
Benzo(k)fluoranthene	8270C	0.01
Benzyl Alcohol	8270C	0.05
4-Bromophenylphenylether	8270C	0.01
Butylbenzylphthalate	8270C	0.01
4-Chloro-3-methylphenol	8270C	0.01
4-Chloroaniline	8270C	0.01
Chlorbenzilate	8270C	0.05
bis(2-Chloroethoxy)methane	8270C	0.01
bis(2-Chloroethyl)ether	8270C	0.01

TABLE D-5 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
bis(2-Chloroisopropyl)ether	8270C	0.01
Bis(2-ethylhexyl)phthalate	8270C	0.01
2-Chloronaphthalene	8270C	0.01
2-Chlorophenol	8270C	0.01
4-Chlorophenylphenylether	8270C	0.01
Chrysene	8270C	0.01
Diallate	8270C	0.05
Dibenzofuran	8270C	0.01
Dibenz(a,h)anthracene	8270C	0.01
3,3'-Dichlorobenzidine	8270C	0.02
2,4-Dichlorophenol	8270C	0.010
2,6-Dichlorophenol	8270C	0.05
Diethylphthalate	8270C	0.010
Dimethoate	8270C	0.050
p-Dimethylaminoazobenzene	8270C	0.050
3,3'-Dimethylbenzidine	8270C	0.02
7,12-Dimethylbenz[a]anthracene	8270C	0.05
2,4-Dimethylphenol	8270C	0.01
Dimethylphthalate	8270C	0.01
a,a-Dimethylphenethylamine	8270C	0.010
Di-n-butylphthalate	8270C	0.01

TABLE D-5 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
4,6-Dinitro-2-methylphenol	8270C	0.025
1,3-Dinitrobenzene	8270C	0.05
1,2-Dinitrobenzene	8270C	0.01
2,4-Dinitrophenol	8270C	0.025
2,4-Dinitrotoluene	8270C	0.01
2,6-Dinitrotoluene	8270C	0.01
Di-n-octylphthalate	8270C	0.01
Dinoseb	8270C	0.01
Diphenylamine	8270C	0.05
Disulfoton	8270C	0.01
Ethylmethane sulfonate	8270C	0.05
Famphur	8270C	0.05
Fluoranthene	8270C	0.01
Fluorene	8270C	0.01
Hexachlorobenzene	8270C	0.01
Hexachlorocyclopentadiene	8270C	0.01
Hexachloroethane	8270C	0.01
Hexachlorophene	8270C	0.01
Hexachloropropene	8270C	0.05
Indeno(1,2,3-cd)pyrene	8270C	0.01
Isodrin	8270C	0.050

TABLE D-5 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
Isophorone	8270C	0.01
Isosafrole	8270C	0.050
Methapyrilene	8270C	0.050
3-Methylcholanthrene	8270C	0.050
Methylmethanesulfonate	8270C	0.05
2-Methylnaphthalene	8270C	0.01
2-Methylphenol	8270C	0.01
3-Methylphenol	8270C	0.01
m,p-Methylphenol	8270C	0.010
Naphthalene	8270C	0.01
1,4-Naphthaquinone	8270C	0.05
1-Naphthylamine	8270C	0.05
2-Naphthylamine	8270C	0.05
2-Nitroaniline	8270C	0.025
3-Nitroaniline	8270C	0.025
4-Nitroaniline	8270C	0.025
Nitrobenzene	8270C	0.01
5-Nitro-o-toluidine	8270C	0.05
2-Nitrophenol	8270C	0.01
4-Nitrophenol	8270C	0.025
4-Nitroquinoline N-oxide	8270C	0.01

TABLE D-5 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
N-nitrosodibutylamine	8270C	0.05
N-nitrosodiethylamine	8270C	0.05
N-nitrosodi-n-propylamine	8270C	0.01
N-nitrosodiphenylamine	8270C	0.01
N-nitrosodimethylamine	8270C	0.01
N-nitrosomethylethylamine	8270C	0.05
N-nitrosomorpholine	8270C	0.01
N-nitrosopiperidine	8270C	0.05
N-nitrosopyrrolidine	8270C	0.05
Pentachlorobenzene	8270C	0.050
Pentachloronitrobenzene	8270C	0.050
Pentachlorophenol	8270C	0.025
Phenacetin	8270C	0.050
Phenanthrene	8270C	0.010
Phenol	8270C	0.010
1,4-Phenylenediamine	8270C	0.010
Phorate	8270C	0.010
2-Picoline	8270C	0.010
Pronamide	8270C	0.050
Pyrene	8270C	0.010
Pyridine	8270C	0.010

TABLE D-5 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
Safrole	8270C	0.050
1,2,4,5-Tetrachlorobenzene	8270C	0.050
2,3,4,6-Tetrachlorophenol	8270C	0.050
Tetraethylpyrophosphate	8270C	0.100
Thionazine	8270C	0.050
o-Toluidine	8270C	0.010
1,2,4-Trichlorobenzene	8270C	0.010
2,4,5-Trichlorophenol	8270C	0.025
2,4,6-Trichlorophenol	8270C	0.010
o,o,o-Triethylphosphorothioate	8270C	0.050
1,3,5-Trinitrobenzene	8270C	0.050

TABLE D-6 - GROUNDWATER PARAMETERS (3 WELLS, APPENDIX IX)

PARAMETER	METHOD	PQL mg/l
Antimony	6010B	0.005
Arsenic	6010B	0.005
Barium	6010B	0.010
Beryllium	6010B	0.004
Cadmium	6010B	0.001
Chromium	6010B	0.005
Cobalt	6010B	0.020
Copper	6010B	0.010
Lead	6010B	0.003
Mercury	7470A	0.0002
Nickel	6010B	0.010
Selenium	6010B	0.005
Silver	6010B	0.005
Thallium	6010B	0.002
Tin	6010B	0.050
Vanadium	6010B	0.020
Zinc	6010B	0.020
Cyanide	9012A	0.010
Sulfide	9030A	0.100

TABLE D-6 (CONT.) -GROUNDWATER PARAMETERS (3 WELLS, APPENDIX IX)

PARAMETER	METHOD	PQL mg/l
2,4-D	8151A	0.005
2,4,5-T	8151A	0.0005
2,4,5-TP (Silvex)	8151A	0.0005
Aroclor 1016	8082	0.0005
Aroclor 1221	8082	0.0005
Aroclor 1232	8082	0.0005
Aroclor 1242	8082	0.0005
Aroclor 1248	8082	0.0005
Aroclor 1254	8082	0.0005
Aroclor 1260	8082	0.0005
Aldrin	8081A	0.00005
a-BHC	8081A	0.00005
b-BHC	8081A	0.00005
d-BHC	8081A	0.00005
g-BHC, Lindane	8081A	0.00005
Chlordane	8081A	0.00005
4,4'-DDD	8081A	0.0001
4,4'-DDE	8081A	0.0001
4,4'DDT	8081A	0.0001

TABLE D-6 (CONT.) -GROUNDWATER PARAMETERS (3 WELLS, APPENDIX IX)

PARAMETER	METHOD	PQL mg/l
Dieldrin	8081A	0.0001
Endosulfan I	8081A	0.00005
Endosulfan II	8081A	0.0001
Endosulfan sulfate	8081A	0.0001
Endrin	8081A	0.0001
Endrin aldehyde	8081A	0.0001
Heptachlor	8081A	0.0001
Heptachlor epoxide	8081A	0.00005
Methoxychlor	8081A	0.0001
Toxaphene	8081A	0.003

TABLE D-6 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
Acetone	8260B	0.010
Acetonitrile	8260B	0.005
Acrolein	8260B	0.010
Acrylonitrile	8260B	0.010
Allyl chloride	8260B	0.010
Benzene	8260B	0.002
Bromodichloromethane	8260B	0.002
Bromoform	8260B	0.002
Bromomethane	8260B	0.002
2-Butanone	8260B	0.002
Carbon disulfide	8260B	0.002
Carbon tetrachloride	8260B	0.002
Chlorobenzene	8260B	0.002
Chloroethane	8260B	0.002
Chloroform	8260B	0.002
Chloromethane	8260B	0.002
Chloroprene	8260B	0.005
1,2-Dibromo-3-chloropropane	8260B	0.002
Dibromochloromethane	8260B	0.002
1,2-Dibromoethane	8260B	0.002
Dibromomethane	8260B	0.002

TABLE D-6 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
1,4-Dichloro-2-butene	8260B	0.002
1,2-Dichlorobenzene	8260B	0.002
1,3-Dichlorobenzene	8260B	0.002
1,4-Dichlorobenzene	8260B	0.002
Dichlorodifluoromethane	8260B	0.002
1,1-Dichloroethane	8260B	0.002
1,2-Dichloroethane	8260B	0.002
1,1-Dichloroethene	8260B	0.002
1,2-Dichloroethene (total)	8260B	0.002
1,2-Dichloropropane	8260B	0.002
cis-1,3-Dichloropropene	8260B	0.002
trans-1,3-Dichloropropene	8260B	0.002
1,4-Dioxane	8260B	0.10
Ethylbenzene	8260B	0.002
Ethyl methacrylate	8260B	0.010
Hexachlorobutadiene	8260B	0.002
2-Hexanone	8260B	0.010
Iodomethane	8260B	0.002
Isobutyl alcohol	8260B	0.010
Methacrylonitrile	8260B	0.005
Methyl methacrylate	8260B	0.005

TABLE D-6 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
4-Methyl-2-pentanone	8260B	0.010
Methylene chloride	8260B	0.002
Pentachloroethane	8260B	0.002
Propionitrile	8260B	0.005
Styrene	8260B	0.002
1,1,1,2-Tetrachloroethane	8260B	0.002
1,1,2,2-Tetrachloroethane	8260B	0.002
Tetrachloroethene	8260B	0.002
Toluene	8260B	0.002
1,2,4-Trichlorobenzene	8260B	0.002
1,1,1-Trichloroethane	8260B	0.002
1,1,2-Trichloroethane	8260B	0.002
Trichloroethene	8260B	0.002
Trichlorofluoromethane	8260B	0.002
1,2,3-Trichloropropane	8260B	0.002
Vinyl acetate	8260B	0.002
Vinyl chloride	8260B	0.002
Xylenes	8260B	0.002

TABLE D-6 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
Acenaphthene	8270C	0.010
Acenaphthylene	8270C	0.01
Acetophenone	8270C	0.010
2-Acetylaminofluorene	8270C	0.050
4-Aminobiphenyl	8270C	0.050
Aniline	8270C	0.025
Anthracene	8270C	0.01
Aramite	8270C	0.01
Benzo(a)anthracene	8270C	0.01
Benzo(a)pyrene	8270C	0.010
Benzo(b)fluoranthene	8270C	0.01
Benzo(g,h,i)perylene	8270C	0.01
Benzo(k)fluoranthene	8270C	0.01
Benzyl Alcohol	8270C	0.05
4-Bromophenylphenylether	8270C	0.01
Butylbenzylphthalate	8270C	0.01
4-Chloro-3-methylphenol	8270C	0.01
4-Chloroaniline	8270C	0.01
Chlorbenzilate	8270C	0.05
bis(2-Chloroethoxy)methane	8270C	0.01
bis(2-Chloroethyl)ether	8270C	0.01

TABLE D-6 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
bis(2-Chloroisopropyl)ether	8270C	0.01
Bis(2-ethylhexyl)phthalate	8270C	0.01
2-Chloronaphthalene	8270C	0.01
2-Chlorophenol	8270C	0.01
4-Chlorophenylphenylether	8270C	0.01
Chrysene	8270C	0.01
Diallate	8270C	0.05
Dibenzofuran	8270C	0.01
Dibenz(a,h)anthracene	8270C	0.01
3,3'-Dichlorobenzidine	8270C	0.02
2,4-Dichlorophenol	8270C	0.010
2,6-Dichlorophenol	8270C	0.05
Diethylphthalate	8270C	0.010
Dimethoate	8270C	0.050
p-Dimethylaminoazobenzene	8270C	0.050
3,3'-Dimethylbenzidine	8270C	0.02
7,12-Dimethylbenz[a]anthracene	8270C	0.05
2,4-Dimethylphenol	8270C	0.01
Dimethylphthalate	8270C	0.01
a,a-Dimethylphenethylamine	8270C	0.010
Di-n-butylphthalate	8270C	0.01

TABLE D-6 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
4,6-Dinitro-2-methylphenol	8270C	0.025
1,3-Dinitrobenzene	8270C	0.05
1,2-Dinitrobenzene	8270C	0.01
2,4-Dinitrophenol	8270C	0.025
2,4-Dinitrotoluene	8270C	0.01
2,6-Dinitrotoluene	8270C	0.01
Di-n-octylphthalate	8270C	0.01
Dinoseb	8270C	0.01
Diphenylamine	8270C	0.05
Disulfoton	8270C	0.01
Ethylmethane sulfonate	8270C	0.05
Famphur	8270C	0.05
Fluoranthene	8270C	0.01
Fluorene	8270C	0.01
Hexachlorobenzene	8270C	0.01
Hexachlorocyclopentadiene	8270C	0.01
Hexachloroethane	8270C	0.01
Hexachlorophene	8270C	0.01
Hexachloropropene	8270C	0.05
Indeno(1,2,3-cd)pyrene	8270C	0.01
Isodrin	8270C	0.050

TABLE D-6 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
Isophorone	8270C	0.01
Isosafrole	8270C	0.050
Methapyrilene	8270C	0.050
3-Methylcholanthrene	8270C	0.050
Methylmethanesulfonate	8270C	0.05
2-Methylnaphthalene	8270C	0.01
2-Methylphenol	8270C	0.01
3-Methylphenol	8270C	0.01
m,p-Methylphenol	8270C	0.010
Naphthalene	8270C	0.01
1,4-Naphthaquinone	8270C	0.05
1-Naphthylamine	8270C	0.05
2-Naphthylamine	8270C	0.05
2-Nitroaniline	8270C	0.025
3-Nitroaniline	8270C	0.025
4-Nitroaniline	8270C	0.025
Nitrobenzene	8270C	0.01
5-Nitro-o-toluidine	8270C	0.05
2-Nitrophenol	8270C	0.01
4-Nitrophenol	8270C	0.025
4-Nitroquinoline N-oxide	8270C	0.01

TABLE D-6 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
N-nitrosodibutylamine	8270C	0.05
N-nitrosodiethylamine	8270C	0.05
N-nitrosodi-n-propylamine	8270C	0.01
N-nitrosodiphenylamine	8270C	0.01
N-nitrosodimethylamine	8270C	0.01
N-nitrosomethylethylamine	8270C	0.05
N-nitrosomorpholine	8270C	0.01
N-nitrosopiperidine	8270C	0.05
N-nitrosopyrrolidine	8270C	0.05
Pentachlorobenzene	8270C	0.050
Pentachloronitrobenzene	8270C	0.050
Pentachlorophenol	8270C	0.025
Phenacetin	8270C	0.050
Phenanthrene	8270C	0.010
Phenol	8270C	0.010
1,4-Phenylenediamine	8270C	0.010
Phorate	8270C	0.010
2-Picoline	8270C	0.010
Pronamide	8270C	0.050
Pyrene	8270C	0.010
Pyridine	8270C	0.010

TABLE D-6 (CONTINUED) - GROUNDWATER PARAMETERS (8 WELLS)

PARAMETER	METHOD	PQL mg/l
Safrole	8270C	0.050
1,2,4,5-Tetrachlorobenzene	8270C	0.050
2,3,4,6-Tetrachlorophenol	8270C	0.050
Tetraethylpyrophosphate	8270C	0.100
Thionazine	8270C	0.050
o-Toluidine	8270C	0.010
1,2,4-Trichlorobenzene	8270C	0.010
2,4,5-Trichlorophenol	8270C	0.025
2,4,6-Trichlorophenol	8270C	0.010
o,o,o-Triethylphosphorothioate	8270C	0.050
1,3,5-Trinitrobenzene	8270C	0.050

TABLE D-6 (CONT.) -GROUNDWATER PARAMETERS (3 WELLS, APPENDIX IX)

PARAMETER	METHOD	PQL mg/l
Kepone	8141A	0.001
Methylparathion	8141A	0.001
Parathion	8141A	0.001
2,3,7,8-Tetrachlorodibenzo-o-dioxin	8280A/8290	10 ppt/10-100 ppq
Polychlorinated dibenzofurans; PCDFs	8280A/8290	10 ppt/10-100 ppq
Polychlorinated dibenzo-p-dioxins; PCDDs	8280A/8290	10 ppt/10-100 ppq